VOL. XXXII 1917

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AMERICAN ELECTROCHEMICAL SOCIETY.

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TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

VOLUME XXXII

THIRTY-SECOND GENERAL MEETING

PITTSBURGH, PA., OCTOBER 3, 4, 5 AND 6, 1917

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1918

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VOLUME XXXII 1917

TRANSACTIONS

OF THE

AMERICAN ELECTROCHEMICAL SOCIETY

PROCEEDINGS

CONDENSED MINUTES OF THE THIRTY-SECOND GENERAL MEETING OF THE SOCIETY, HELD IN PITTSBURGH, PA., OCTOBER 3, 4, 5, AND 6, 1917.

Number of members registered, 164; number of guests registered, 189; total, 353.

PROCEEDINGS OF WEDNESDAY, OCTOBER 3

Registration began at the William Penn Hotel, Pittsburgh, at 10.00 A. M.

After lunch, automobiles left the hotel for the Oakmont Country Club, fifteen miles distant, where an informal assembly was staged, golf provided for those who desired to play, and an informal dinner partaken of in the evening.

At 8.00 P. M. the Publication Committee and board of Directors held their semi-annual meetings.

PROCEEDINGS OF THURSDAY, OCTOBER 4

The session was called to order in the auditorium of the William Penn Hotel, Pittsburgh, by President C. G. Fink, at 10.00 A. M. President Fink introduced Dr. John A. Brashear, a well-known Pittsburgh scientist and philanthropist, who made a lengthy address of welcome, discussing the various private and public industries and institutions for which Pittsburgh is famous. He referred to the contribution of Pittsburghers to science, alluded to the fact that for thirty years he was associated with the work of Professor Samuel P. Langley; also to the work of the astronomer Keeler, of the Engineer Westinghouse, and to the beginning of the aluminum industry in Pittsburgh. Thirty-five years ago Dr. Brashear paid $2.50 per ounce for some alumi-

num to Frischmuth, of Philadelphia, and now it can be bought for less than forty cents per pound, thanks to the works in Pittsburgh. Amongst the educational advantages of Pittsburgh, Dr. Brashear mentioned the University of Pittsburgh with 3,700 students, the Carnegie Institute of Technology with 3,600 students, the Carnegie Library with its four branches, circulating over two million books per year; the eighteen city parks; the Mellon Institute of Industrial Research—a model of similar institutions now being founded in different parts of the world.

The chairman next called upon Mr. Lawrence Addicks, a representative of the Society upon the Naval Consulting Board, to speak of the work of the Board during the past year. Mr. Addicks spoke as follows:

"It is quite obvious that I cannot tell you just what has been done, and I can only tell you a word about how it has been done. I think the Board has been more fortunate than some other official bodies in Washington, in that it has had the definite problems and definite responsibility of one of the Cabinet Officers. It has been able to work more consistently and rapidly than some other bodies not so fortunately placed. Both of your representatives on the Board have been busy.

"Dr. J. W. Richards, our other representative, has been giving all of his time during the past summer to reviewing the subjects that come in from all parts of the country, and I am chairman of the Committee on Special Problems, which deals with what you might call the internal side, that is, organizing among the members themselves, and a few associates chosen by the members, to see what application could be made of these to the solution of our new problems.

"On the internal side, all of the members of the Board realize they are representatives of the bodies they represent. They wish to work back to the membership as a whole. That has been difficult to do as a whole, as publicity comes in.

"To meet the exigency we got together a limited number of men to whom the problems would be made clear. Each member of the Board was asked to nominate ten men with special qualifications, men whose loyalty we could vouch for. From that special list it dwindled down to thirty men. Early in March we met in New York with Captain Simms, now Vice-Admiral

abroad, and some officers who are experts on some of the technical matters. The whole topic was developed for plans for the detection of submarines, of sound methods, microphones, and magnetic and optical methods. The naval officers answered all questions, and after the meeting sub-committees were formed and laboratory work was started. One was started on the Atlantic Coast to go into the methods of sound detection; another was started on magnetic and electrical methods of detection. There has been nothing miraculous obtained, but there is progress in these new problems, and I hope this progress will solve the problem of detecting and annihilating the submarine.

"That is about all I can think of in the two or three minutes I have, that I feel at liberty to mention."

Secretary J. W. Richards announced to the meeting some actions taken by the Board of Directors at its meeting the evening before, such as an appropriation of $2,000 to be invested in Liberty Bonds and the recommendation of holding a meeting of the Society in the Appalachian South in the near future if it was practicable.

Reading and discussion of papers was then taken up, and papers as follows were considered: Henry Randall, J. L. McK. Yardley, Robert Turnbull, R. E. Lowe, Frank Thornton, Jr., Haakon Styri, T. F. Baily, T. D. Yensen, E. D. Campbell, and O. L. Kowalke (read by title). These papers and their discussions are printed in full in these Transactions.

Luncheon was taken in the hotel, being complimentary to visitors and guests from the Pittsburgh Local Section.

At 2.00 P. M. six alternative visits were provided, one by special train to the American Zinc and Chemical Co. of Langloth, Pa., another to the Research Laboratory of the American Sheet and Tin Plate Co., another to the Mellon Institute of Industrial Research, another to the University of Pittsburgh, another to the Carnegie Technical School, and one to the Carnegie Museum.

In the evening, at 8.00 P. M., Mr. Alex Dow, President of the Detroit Edison Company, delivered an address in the Banquet Hall of the William Penn Hotel, on the "Production of Electricity by Steam Power," which was followed by general discussion of the power problem. The address and discussion are printed in full in these Transactions.

Later in the evening an informal reception was held, at which music, dancing and refreshments were indulged in, the Pittsburgh Local Section being the hosts.

PROCEEDINGS OF FRIDAY, OCTOBER 5

The meeting was called to order at 10.00 A. M. by President C. G. Fink, in the ballroom of the William Penn Hotel. Papers were read and discussed as follows: Carl Hering, C. H. Viol and G. D. Kammer (presented by Mr. Kammer), W. D. Marshall (read by title), L. B. Cherry, C. W. Hill and G. P. Luckey (presented by Mr. Hill), G. F. Comstock (abstracted by Secretary Richards), J. L. Jones (two papers), L. C. Turnock, O. W. Storey, J. Coulson, T. S. Fuller, O. P. Watts and N. D. Whipple (read by title), W. D. Bancroft (three papers), Thos. French. These papers and their discussions are printed in full in these Transactions.

In the afternoon six alternative trips had been arranged, as follows: The Benzol and By-product Coke Plants of the Briar Hill Steel Co., Youngstown, Ohio; New Laboratory of the Youngstown Sheet and Tube Co.; The Atlantic Refining Co. Oil Works; The Owens Bottle Works, Sharpsburg, Pa.; The Republic Chemical Co. Detinning Plant, Neville Island; The Radium Research Laboratory of the Standard Chemical Co.

In the evening a subscription dinner was participated in by about 200 members and guests, in the grand ballroom of the William Penn Hotel. Entertainment features were liberally provided, and the evening was most enjoyably spent.

PROCEEDINGS OF SATURDAY, OCTOBER 6

This day was devoted entirely to a train excursion on special train, with complimentary luncheon, through several of the most interesting manufacturing plants of the Pittsburgh district. Starting early in the morning the following plants were visited during the day: Westinghouse Electric and Manufacturing Co. plant at East Pittsburgh; National Tube Company plant at McKeesport; the Duquesne works of the Carnegie Steel Co., where the largest electric furnaces in America were seen in operation; the American Window Glass Company plant at Arnold.

The whole excursion was admirably arranged and managed and reflected great credit on the Pittsburgh Local Section. A

souvenir program (of fifty pages) of the meeting, admirably written and illustrated, provided with maps, etc., described the various visits and entertainments for the members and guests and those arranged for the ladies in attendance, and added very much to the pleasure and profit of those taking part in the meeting.

At the closing session the appreciation of the visiting members was expressed in a hearty vote of thanks to the industrial concerns who had so liberally opened their works, the local members and their friends who had so hospitably entertained the visitors, and the members of the Local Section who had given so unsparingly of their time and substance toward the success of the meeting.

MEMBERS AND GUESTS REGISTERED AT THE THIRTY-SECOND ANNUAL MEETING.

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Walter Arthur
James Aston
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A. T. Baldwin
W. D. Bancroft
H. L. Barr
E. R. Berry
E. L. Blossom
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H. R. Connell
F. T. Cope
Fred Crabtree
John Cranston
C. A. Crawford
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H. T. Darlington
J. V. N. Dorr
J. W. Dougherty
A. C. Downes
Boyd Dudley, Jr.
A. L. Duval
F. F. Farnham
C. G. Fink
F. A. J. FitzGerald
J. A. Fogarty
C. L. Foster
H. W. Fox
F. C. Frary
R. H. Gaines
C. B. Gibson
A. McK. Gifford
H. W. Gillett
C. A. Gillingham
G. C. Given
S. L. Goodale
G. M. Goodspeed
I. Gotoh
V. C. Hamister
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E. S. Hedstrom
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C. F. Hirshfeld
H. K. Hitchcock
B. H. Hite
G. B. Hogaboom
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W. F. James
E. G. Jarvis
G. Jinguji
J. F. Kelly
Philo Kemery
F. L. Koethen
H. L. Kohler

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Walter Laib
F. A. Lidbury
P. M. Lincoln
M. G. Lloyd
G. O. Loeffler
E. A. Lof
R. E. Lowe
D. A. Lyon
J. D. MacMahon
C. P. Madsen
J. L. Malm
C. A. Mann
P. Martens
J. A. Mathews
D. L. Mathias
G. G. Mattern
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G. P. McNiff
A. Merzbacher
H. H. Meyers
D. D. Miller
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R. W. E. Moore
W. R. Mott
J. M. Muir
Y. Nakamura
S. Nakashima
J. R. H. Neal
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Chas. Wirt
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F. C. Woodside
J. L. M. Yardley
T. D. Yensen
J. A. Yunck
R. E. Zimmerman

Guests.

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E. Arakawa, Kyoto, Japan
J. E. Baff, Pittsburgh, Pa.
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C. A. Basore, Pittsburgh, Pa.
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E. E. Boon, East Pittsburgh, Pa.
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Catharine Burns, Lancaster, Pa.
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Harry Essex, Pittsburgh, Pa.
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Miss Ford, Pittsburgh, Pa.
J. L. Foster, Jr., Pittsburgh, Pa.
J. H. Fox, Pittsburgh, Pa.
M. Fukuda, Tokyo, Japan.
T. S. Fuller, Schenectady, N. Y.
K. H. Fulton, Pittsburgh, Pa.
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G. F. Gray, Pittsburgh, Pa.
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Mrs. S. G. Haffal, Aliquippa, Pa.
Henry Hagel, Dravosburg, Pa.
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ABBE, Paul O. (Nov. 27, '09) res., 4434 18th Ave., Brooklyn, N. Y.; mailing address, 30 Broad St., New York City.
ABBOTT, William G., Jr. (Mar. 27, '09) care of Hillsboro Mills, Wilton, N. H.
ACHESON, E. G. (Apr. 3, '02) Acheson Corp., Aeolian Bldg., 35 W. 42d St., New York City.
ACKER, Charles E. (Apr. 3, '02) President, The Nitrogen Co., Ossining-on-Hudson, N. Y.
ACTON, E. H. (Oct. 23, '14) Supt. Northern Aluminum Co., Shawinigan Fall, P. Q., Canada.
ADAMS, James L., Jr. (June 2, '16) 420 Montview Place, Wilkinsburg, Pa.
ADAMS, Quinton (July 28, '16) Elec. Eng., 1st National Bank Bldg., 1 Montgomery St., San Francisco, Cal.
ADAMS, Thos. J. (Aug. 25, '16) Res. Asst., Aluminum Co. of America, Maryville, Tenn.
ADAMSON, G. P. (Dec. 4, '02) Gen. Mgr., Baker & Adamson Chem. Co.; res., 233 Reeder St., Easton, Pa.
ADDICKS, Lawrence (Apr. 3, '02) Consulting Eng., Room 2, 126 Liberty St., New York City.
ADSIT, Charles G. (Sept. 26, '08) 1 Ponce de Leon Ave., Atlanta, Ga.
AHLBRANDT, G. F. (Jan. 29, '09) Asst. Gen. Sales Mgr., Amer. Rolling Mills Co., Middletown, Ohio.
AIKEN, Robert H. (Oct. 10, '03) Metallurgist, Winthrop Harbor, Ill.
AKINEIEFF, Boris (Oct. 26, '17) Inspector, Russian Artillery Commission; mailing address, Pine Terrace Inn, Dover, N. J.
ALBRIGHT, Langdon (Sept. 26, '08) Vice-Pres., Niagara, Lockport and Ont. Power Co., 1608 Marine Bank Bldg., Buffalo, N. Y.; res., 33 Oakland Place.
ALDRICH, Chas. H. (July 25, '13) Metallurgist, Colgate, Baltimore County, Md.
ALDRIDGE, Walter H. (Jan. 28, '08) 14 Wall St., New York City.
ALEXANDER, C. M. (Sept. 27, '16) Res. Chemist, Gulf Ref. Co., Port Arthur, Texas.
ALEXANDER, H. H. (July 25, '13) Mgr., American Smelt. and Ref. Co., Perth Amboy, N. J.; res., Maurer, N. J.
ALLEMAN, Gellert (Jan. 28, '11) Professor of Chemistry, Swarthmore College, Swarthmore, Pa.
ALLEN, Herbert I (July 25, '13) 286 State St., Portland, Me.
ALLEN, O. F. (Oct. 21, '16) Sales Eng., General Elec. Co., Room 1712, 30 Church St., New York City.
ALLYN, R. S. (Feb. 5, '03) Patent Lawyer, 16 Exchange Place, New York City, res., 24 Irving Place, Brooklyn, N. Y.
AMBERG, Dr. Richard (Mar. 26, '10) Schwerte i. W., Germany.
AMER, Henry S. (May 27, '11) Assistant in Eng. Dept., Westinghouse Lamp Co., Bloomfield, N. J.; mailing address, 14 Evergreen Ave.
AMINOFF, Gustav (Feb. 27, '09) Abo Vuoksenniska (Finland) Russia.
ANDERSON, Chas. E. (Sept. 27, '16) Gen. Mgr., Smuggler Leasing Co., Aspen, Colo.
ANDREWS, Joseph C. (May 26, '10) Chief Chemist, American Hardware Corp., New Britain, Conn.; mailing address, 123 Vine St.
ANDREWS, J. H. (May 2, '17) Electroplater, Dunn and Rodenbery; mailing address, 146 Hanover St., Providence, R. I.
ANDREWS, Miss Mary R. (Feb. 24, '17) Experimental Biological Dept., Rockefeller Inst., 66th St. and Ave. A., New York City.
ANDREWS, Wm. S. (April 6, '11) Consulting Eng., General Electric Co., Schenectady, N. Y.; res., 136 Park Ave.
ANGER, Earl M. (May 25, '17) 514 W. 135th St., New York City.
ANTISELL, Frank L. (July 25, '13) Asst. Supt., Raritan Copper Works, Perth Amboy, N. J.

ANTUNEZ, Vicente (June 30, '16) Miner, 325 Merced, Santiago, Chile.
ARISON, William H. (July 31, '08) Mgr., Acheson Graphite Co., Niagara Falls, Ont., Canada.
ARMES, Henry P. (Jan. 27, '12) Lecturer in Chemistry, University of Manitoba, Manitoba, Winnipeg, Canada.
ARMSTRONG, Lyndon K. (Aug. 27, '09) Mining Engineer, Spokane, Washington; mailing address, P. O. Drawer 2154, Spokane, Wash.
ARNEMANN, Dr. Paul (Apr. 29, '11) Grindelhof 19, Hamburg 13, Germany.
ARSEM, William C. (Nov. 27, '09) Chemical Engineer, General Electric Co., Schenectady, N. Y.; res., 10 Waverly Place.
ARTHUR, Paul (Jan. 26, '17) Mech. Eng., Simms Magneto Co.; mailing address, 12 N. Arlington Ave., East Orange, N. J.
ARTHUR, Walter (Apr. 27, '12) Chief Chemist, Garford Motor Truck Co., Lima, Ohio.
ASEF, Dr. Waldemar (Oct. 21, '16) Res. Chem., Penna. Salt Mfg. Co., Greenwich Point, Philadelphia.
ASHCROFT, Edgar A. (May 1, '06) Consult. Eng. and Res. Chem., 65, London Wall, London, E. C., England.
ASKENASY, Paul, Ph. D. (Feb. 25, '11) Kaiser Allee 20, Karlsruhe (Baden), Germany.
ASTON, James (Jan. 28, '08) 50 Forest Ave., Ben Avon, Pa.
ATWATER, C. G. (Oct. 22, '15) Mgr. of Agricultural Dept., The Barrett Co.; 17 Battery Place, New York City.
ATWOOD, F. C. (Sept. 27, '16) Res. Eng., The Exolon Co., Thorold, Ont., Canada.
AUSTIN, A. O. (Sept. 27, '16) Chief Eng., The Ohio Insulator Co.; mailing address, 326 North Sixth St., Barberton, Ohio.
AUSTIN, L. S. (Sept. 27, '16) 251 West 2d North St., Salt Lake City, Utah.
BABROWSKY, G. (Mar. 25, '08) Inst. for Theor. and Physical Chemistry, Ceská vysoká skola technika, Brünn, Moravia, Austria.
BACHOFNER, D. K. (Mar. 26, '10) Assistant Superintendent, Irvington Smelting and Refining Co., Irvington, N. J.
BACON, R. F. (Nov. 28, '13) Director Mellon Inst., Univ. of Pittsburgh, Pittsburgh, Pa.
BADGER, W. L. (Feb. 24, '17) Asst. Prof. of Chem. Eng., Univ. of Michigan; mailing address, 1317 Minerva Road, Ann Arbor, Mich.
BAEKELAND, Dr. Leo H. (Jan. 6, '03) "Snug Rock," Harmony Park, Yonkers-on-Hudson, N. Y.
BAILEY, Richard O. (Oct. 21, '16) Chemist, Oneida Community Ltd., Oneida, N. Y.
BAILY, Thaddeus F. (Feb. 25, '11) Gen. Mgr., The Electric Furnace Co. of America, Alliance, Ohio.
BAIN, J. Watson (Oct. 27, '11) Prof. of Chemical Engineering, C. & M. Bldg., The University, Toronto, Canada.
BAJDA, Jas. J. (Sept. 27, '16) 211 Murray St., Flushing, N. Y.
BAKER, Chas. E. (Aug. 5, '05) Metallurgist, 201 Traders Bldg., Chicago, Ill.; mailing address, 324 Postal Bldg., La Salle and Van Buren Sts., Chicago, Ill.
BAKER, E. M. (Apr. 26, '16) Chemist, Hooker Electrochemical Co., Niagara Falls, N. Y.; mailing address, 351 Sixth St.
BAKER, Herbert A. (Dec. 27, '07) Chemist, care of the American Can Co., 447 W. 14th St., New York City.
BAKER, John T. (Mar. 30, '08) Phillipsburg, N. J.
BAKER, L. R. (Mar. 24, '16) Mgr., Canada Dry Cells Ltd.; mailing address, Oakview Ave., East Kildonan, Manitoba, Canada.
BAKER, Robt. E. (Jan. 22, '15) Wks. Mgr.; Keokuk Electro-Metals Co.; mailing address, 526 N. 4th St., Keokuk, Iowa.
BALDWIN, Allen T. (Dec. 31, '15) Chem., Manhattan Elec. Supply Co., 41-47 Morris St., Jersey City, N. J.
BALTZELL, Willie H. (May 27, '11) Canadian Steel Corp. Ltd., Ojibway, Ontario, Canada.
BANCROFT, Wilder D., Ph.D. (Apr. 3, '02) Cornell University; res., 7 East Ave., Ithaca, N. Y.
BARCLAY, Earle H. (Oct. 21, '16) 934 Holladay St., Portsmouth, Va.
BARFOED, Svend (Aug. 25, '17) Engr. in charge of Experiments, Nitrogen Products Co.; mailing address, 908 Insurance Exchange Bldg., San Francisco, Cal.
BARKER, E. R. (Apr. 3, '02) 79 Milk St., Room 1110, Boston, Mass.
BARNES, H. H., Jr. (June 30, '16) District Eng., General Elec. Co., 120 Broadway, New York City.
BARNES, W. A. (Apr. 26, '13) Clift Hotel, San Francisco, Cal.
BARNHILL, W. J. (Sept. 27, '16) Owner Chem. Labs.; mailing address, P. O. Box 306, Colton, Cal.
BARR, Harry L. (Oct. 21, '16) Chem. and Asst. Electrochem., Follansbee Bros. Co., 1075 Virginia Ave., Follansbee, W. Va.
BARROWS, F. E. (Aug. 22, '13) Patent Attorney, 35 Nassau St., New York City.

BARROWS, W. S. (Apr. 26, '13) Foreman Electro-plater, Russell Motor Car Co.; res., 628 Dovercourt Road, Toronto, Ont., Canada.
BARSTOW, W. S. (Apr. 3, '02) 56-58 Pine St., New York City.
BARTELL, F. E. (Apr. 26, '17) Ass't Prof., Chem. Lab. Univ. of Mich., Ann Arbor, Mich.
BARTON, Charles B. (Sept. 4, '02) Brown Co., Berlin, N. H.
BARTON, P. P. (Feb. 2, '06) Gen. Mgr., Niagara Falls Power Co., 352 Buffalo Ave., Niagara Falls, N. Y.
BARTON, W. H. (Feb. 25, '11) Met. Chemist, Searchlight, Nev.
BARWICK, William S. (Dec. 31, '09) Chemist and Metallurgist, Vancouver Engineering Works, Vancouver. B. C., Canada; mailing address, 1357 Barclay St.
BASKERVILLE, Chas., Ph.D. (Apr. 4, '03) Prof. of Chem., College City of New York, New York City.
BASSETT, Wm. H. (Nov. 26, '07) Technical Supt. and Metallurgist, The American Brass Co., Waterbury. Conn.; res., Cheshire, Conn.
BATCHELDER, John K. (Apr. 2, '13) Supt. Lead and Copper Ref., Consolidated Mining and Smelting Co. of Canada, Ltd., Trail, B. C., Canada.
BATE, H. A. (Sept. 27, '16) Eng. of Tests, Ordnance Dept., U. S. Government, c|o Bartlett-Hayward Co., Baltimore, Md.
BAUER, G. W. (Sept. 4, '03) Vice-Pres. and Chem., Bauer, Schweitzer Hop and Malt Co., 1722 Buchanan St., San Francisco, Cal.
BAUER, Wm. Chas. (May 27, '11) Professor of Electrical Engineering, Northwestern University, Evanston, Ill.; res., 2149 Sherman Ave., Evanston, Ill.
BAYARD, R. A. (Apr. 24, '14) Resident Supt., Chippewa Plants, Norton Co., Chippewa, Ont.; mailing address, 170 Buffalo Ave., Niagara Falls, N. Y.
BEAR, S. L. (Feb. 26, '15) Gen. Eng., West. Elec. & Mfg. Co., 4001 Northminster St., North Side, Pittsburgh, Pa.
BEARD, C. W. (Jan. 26, '17) Chemist, Niagara Alkali Co., 21 Mentz Apts., Niagara Falls, N. Y.
BECK, Dr. Erick A. (Apr. 29, '11) Metallurgist, Goldschmidt Thermit Co., 120 Broadway, New York City; res., 931 Park Ave.
BECKET, Frederick M. (Apr. 3, '02) The El.-Met. Co. of America, 403 Jefferson Ave., Niagara Falls, N. Y.
BECKMAN, John Woods (May 6, '05) Beckman & Linden Engineering Corp., Balboa Bldg., San Francisco, Cal.; mailing address, 203 Hillcrest Road, Berkeley, Cal.
BEDELL, E. H. (Oct. 22, '15) Advertising Solicitor, McGraw-Hill Pub. Co., 10th Ave. and 36th St., New York City.
BENFIELD, Bernard (Oct. 3, '17) Consult. Eng. in Petroleum and Asphaltum; mailing address, 719 Rialto Bldg., San Francisco, Cal.
BENJAMIN, E. O. (Oct. 26, '17) Chief Chem. and Eng., International Oxygen Co.; mailing address, 177 Hillside Ave., Newark, N. J.
BENNETT, M. H. (Nov. 28, '13) Res. Engineer, 26 Linden St., Waterbury, Conn.
BENNIE, P. McN. (July 1, '04) 500 Queen St., East, Toronto, Ont., Canada.
BENOLIEL, Sol. D., B.S., E.E., A.M. (Sept. 4, '02) Gen. Mgr. International Chem. Co., Camden, N. J.; mailing address, 4508 Locust St., Philadelphia, Pa.
BERGEN, R. C. (Oct. 22, '15) Asst. Editor, Met. and Chem. Eng., 10th Ave. and 36th St., New York City.
BERGLUND, Edw. S. (Aug. 30, '12) Manager and Chief Engineer, Trollhättan Elekthermiska Aktiebolag, Trolhättan, Sweden.
BERRY, Edw. R. (Dec. 1, '06) Chief Chem., Eng. Lab., Gen. Elec. Co., Lynn, Mass.; res., 30 Hancock St., Malden, Mass.
BERTHIER, U. H. (Sept. 27, '16) c|o Cia Minera Palomay Cabrillas S. A., Higueras, Coahuila, Mexico.
BETTS, Anson G. (Apr. 3, '02) care of Anson G. Betts & Co., 519 Legal Bldg., Asheville, N. C.
BIERBAUM, Christopher H. (Apr. 3, '02) Consulting Engineer, 1011 Mutual Life Bldg., Buffalo, N. Y.
BIGELOW, S. Lawrence, Ph.D. (May 9, '03) Prof. of Gen. and Phys. Chem., Univ. of Mich.; res., 1520 Hill St., Ann Arbor, Mich.
BIJUR, Joseph (Sept. 19, '03) Bijur Motor Lighting Co., River Front and 15th Sts., Hoboken, N. J.; mailing address, Garden and 15th Sts.
BJÖRKSTEDT, Wm. (Apr. 6, '11) Electrometallurgical Engineer, 1113 Walnut St., Wilkinsburg, Pa.
BJORNSON, Einar (Oct. 29, '10) Arbins Gt. 13, Christiania, Norway.
BLANCHARD, H. J. (Nov. 24, '16) Chemist, Titanium Bronze Co., Niagara Falls, N. Y.; mailing address, 722 7th St.
BLASIUS, C. E. (May 2, '17) Student, Lehigh Univ., Bethlehem, Pa.; mailing address, Phi Gamma Delta House, 414 Cherokee St.
BLEEKER, Warren F. (Nov. 27, '09) Boulder, Colo.
BLISS, Wm. L. (Mar. 23, '12) Chief Engineer, U. S. Light and Heating Co.; res., 142 Buffalo Ave., Niagara Falls, N. Y.
BLOSSOM, E. L. (Nov. 23, '17) 701 Kearns Bldg., Salt Lake City, Utah.

BLOUGH, Earl (Apr. 29, '11) Chief Chemist, Aluminum Co. of America, Parnassus, Pa.
BLUM, William (Nov. 26, '15) Associate Chemist, Bureau of Standards, Washington, D. C.
BOECK, Percy A. (Feb. 28, '08) Eastern District Mgr., Celite Products Co., 11 Broadway, New York City; mailing address, W. 227th St. and Netherland Ave., New York City.
BOERICKE, Gideon (Mar. 5, '04) Sec. and Treas., Primos Chem. Co., Primos, Delaware County, Pa.
BOESCH, John E. (Aug. 27, '09) E. E. & M. E., Cerro de Pasco Mining Co., Lima, Peru.
BOGUE, Chas. J. (Apr. 3, '02) Mfr. of Elec. Machinery, 213-215 Center St., New York City.
BOICE, Edwin N. (June 1, '15) Treasurer, care of The Hanson & Van Winkle Co., 269 Oliver St., Newark, N. J.
BONINE, Chas. E. (Aug. 30, '12) Cons. Engineer, 610 Harrison Bldg., Philadelphia, Pa.; res., 4153 Leidy Ave.
BONNEVIE, Harald (Dec. 31, '14) Chief Engr. and Mgr. of Rjukan Saltpeterverk, Rjukan, Norway.
BONSIB, R. S. (Oct. 21, '16) Res. and Exp. Eng., U. S. Metals Ref. Co., East Chicago, Ind.
BOOTH, W. K. (Aug. 25, '16) Gen. Mgr., Midland Electric Steel Co., Terre Haute, Indiana.
BORN, Sidney (Aug. 25, '17) Chief Chem. and Supt., Cracking Dept., Empire Refineries, Inc.; mailing address, Drawer F, Bartlesville, Okla.
BOSWELL, Walter O. (April 26, '13) Elec. Eng., Engineering Dept. Hydro-Electric Power Commission of Ont.; mailing address, 25 Roxboro St., W., Toronto, Ont., Canada.
BOVEE, Ben. A. (Feb. 26, '15) Instructor, Eng. Dept., School of Engineering; mailing address, 917 4th St., Milwaukee, Wis.
BOWLES, Richard H. (Dec. 30, '16) Asst. E. E., Sao Paulo Tramway, Light and Power Co., Sao Paulo, Brazil.
BOWMAN, C. H. (Dec. 31, '09) President, Montana State School of Mines, Butte, Mont.; mailing address, 1020 Caledonia St.
BOWMAN, Francis C. (Jan. 29, '10) Valley, Washington.
BOWMAN, Walker (Apr. 3, '02) 39 Cortland St., New York City.
BOYNTON Wm. H. (Mar. 26, '15) Analytical Chemist, Crown Cork and Seal Co., Baltimore, Md.; mailing address, 1801 Linden Ave., Baltimore, Md.
BRADFORD, Robert H. (Feb. 25, '11) Professor of Metallurgy, University of Utah, Salt Lake City, Utah.
BRADLEY, D. H., Jr. (Nov. 24, '11) Consulting Engineer, Prescott, Arizona.
BRADLEY, J. C. (Nov. 26, '07) Metallurgist, care of American Brass Co., Waterbury, Conn.; mailing address, Fairlawn Ave.
BRADLEY, Linn (June 30, '16) Chief Eng., Res. Corp.; mailing address, 63 Wall St., New York City.
BRADLEY, Walter E. F. (June 29, '07) Woolworth Bldg., New York City.
BRADLEY, Walter M. (Apr. 3, '02) Sheffield Scientific School, Mineral Lab.; res., 520 Whitney Ave., New Haven, Conn.
BRADSHAW, Hamilton (Jan. 26, '17) Director, Experiment Station, Henry Clay, Delaware.
BRADY, William (May 29, '09) Chief Chemist, Illinois Steel Co., Chicago, Ill.; mailing address, 7642 Marquette Ave.
BRAGG, E. B. (Feb. 25, '11) Manager, Chicago Branch, General Chemical Co., Evanston, Ill.; res., 1838 Chicago Ave.
BRANDT, M. F. (Aug. 25, '16) Industrial Chem., with Francis I. du Pont; mailing address, 109 W. 10th St., Wilmington, Del.
BRAY, William C. (May 30, '08) Dept. of Chemistry, University of California, Berkeley, Cal.
BRECKENRIDGE, C. E. (Oct. 22, '15) Jefferson Ave., Rahway, N. J.
BRECKENRIDGE, J. E. (Apr. 26, '13) Chief Chemist, Am. Agric. Chem. Co., Carteret, N. J.
BREED, George (Jan. 3, '04) Consult. Eng., Witherspoon Bldg., Philadelphia, Pa.
BRENEMAN, A. A. (May 1, '06) Analyt. and Consult. Chem., 97 Water St., New York City.
BRIGGS, Frank H. (Oct. 27, '11) Vice-Pres. and Gen. Mgr., American Dynalite Co., Amherst, Ohio.
BRIGHT, Arthur C. (July 27, '17) Student, Sigma Phi Epsilon House, State College, Pa.
BRINDLEY, Geo. F. (Apr. 3, '02) P. O. Box 202, Wilmington, Del.
BRISTOL, Wm. H. (Dec. 1, '06) Pres., The Bristol Co., Waterbury, Conn.
BROCKWAY, John P. (Sept. 27, '16) 209 Equitable Bldg., Denver, Colo.
BROOKFIELD, Wm. Bertin (May 26, '10) Superintendent, Melting Department, Halcomb Steel Co., Syracuse, N. Y.; res. 868 Ostrom Ave.
BROOKS, Morgan, Ph.D., M.E. (Apr. 3, '02) Univ. of Illinois, Urbana, Ill.

BROOKS, W. C. (Mar. 23, '12) Asst. Supt., National Carbon Co.; mailing address, Suite 4, 1448 Highland Ave., Lakewood, Ohio.

BROPHY, Oscar (Feb. 27, '14) Chem. Engineer, Ajax Metal Co., Philadelphia, Pa.; mailing address, 1919 N. 12th St.

BROWN, C. J. (Oct. 22, '15) Secretary, Wilson-Maeulen Co.; mailing address, 15 Union Ave., Mt. Vernon, N. Y.

BROWN, Harold P. (Apr. 3, '02) Elec. Eng., 120-122 Liberty St., New York City.

BROWN, John T., Jr. (May 26, '10) Superintendent, Duquesne Reduction Co., Pittsburgh, Pa.; res., 5448 Stanton Ave.

BROWN, Dr. John W. (July 31, '07) Pres. and Mgr., The Cleveland Electric Metals Co., Forest City Bank Bldg., Cleveland, Ohio.

BROWN, M. J. (Dec. 31, '15) Chief Chem., Roessler & Hasslacher Chem. Co., Plant DAB, Perth Amboy, N. J.

BROWN, O. W. (Apr. 3, '02) Associate Prof. in Chem., Indiana Univ., Bloomington, Ind.; res. 420 E. 3d St.

BROWN, Richard P. (Aug. 25, '16) Pres., The Brown Instrument Co., Wayne and Windrim Avenues, Philadelphia, Pa.

BROWN, W. G., B.S., Ph.D. (Apr. 3, '02) Prof. of Chem., Univ. of Missouri, Columbia, Mo.

BROWNE, deCourcy B (Apr. 26, '13) Captain U. S. R., Ordnance Dept., American Expeditionary Forces.

BROWNE, Prof. Wm. Hand, Jr. (Apr. 3, '02) North Carolina College of Agriculture and Mechanic Arts, West Raleigh, N. C.

BROWNLEE, Harold J. (Dec. 31, '15) Redmand Chem. Products Co., 636 W. 22d St., Chicago, Ill.

BRUMBAUGH, A. K. (Jan. 26, '17) Asst. Eng., Autocar Co., Ardmore, Pa.

BRYAN, John K. (Aug. 22, '13) care of Electrolytic Zinc Co., Colgate, Baltimore County, Md.

BRYN, Knud (Nov. 24, '11) Director, Kristiania, Norway.

BRYSON, T. A. (Oct. 22, '15) Eng., Tolhurst Steel Co., Troy, N. Y.; mailing address, 2228 15th St.

BUCH, N. W. (Nov. 6, '13) Mgr. P. M. Evans Co., 3707 Ludlow St., Philadelphia, Pa.

BUCHANAN, Leonard B. (Apr. 3, '02) Head of Chemical and Industrial Dept., Stone & Webster, 147 Milk St., Boston, Mass.

BUCHERER, Prof. Dr. Alfred H. (May 27, '11) Professor of Physics, Bonn University, Bonn, Germany.

BUCK, C. A. (May 29, '09) Vice-President, Bethlehem Steel Co., Bethlehem, Pa.

BUCK, H. W. (May 7, '04) 49 Wall St., New York City.

BUCKIE, Robert H. (Sept. 25, '09) care of New York & Penna. Co., Johnsonburg, Pa.

BUHL, Wm. (May 25, '17) E. E., 1811 Washington, St., Easton, Pa.

BULLOCK, Arthur R. (Apr. 24, '14) Electrical Eng., 11913 Lake Ave., Lakewood, Cleveland, Ohio.

BUMGARDNER, J. W. (Aug. 25, '16) Metallurgist, and Supt. Chill Roll Dept., Wheeling Mold and Foundry Co., Wheeling, W. Va.; mailing address, 4 19th St., Warwood, W. Va.

BURDICK, E. C. (Oct. 21, '16) Chemist, Dow Chem. Co., Midland, Mich.

BURGER, Dr. Alfred (Aug. 26, '10) P. O. Box 314, Newark, N. J.

BURGESS, C. F. (Apr. 3, '02) President, C. F. Burgess Labs., 625 Williamson St., Madison, Wis.

BURGESS, Louis (May 2, '17) care of Standard Oil Co., Bayonne, N. J.

BURKE, Wm. E. (Aug. 25, '17) Instructor in Chem., Stanford University, Cal.

BURMAN, Birger F. (Dec. 31, '14) 1717 N. 53d St., Philadelphia, Pa.

BURNS, Willis T. (Nov. 6, '03) Supt., Electrolytic Refinery, Boston and Mont. Cons. Cop. S. and Min. Co., Great Falls, Mont.

BURT, G. G. (May 2, '17) Foreman Plater, 15517 Center Ave., Harvey, Ill.

BURT, Milo C. (Oct. 3, '17) Director, Exp. Lab., care of Atlas Powder Co., Tamaqua, Pa.

BURT-GERRANS, J. T. (Jan. 27, '12) Lecturer in Electrochemistry, University of Toronto, Toronto, Canada; res., 46 Dewson St.

BURWELL, Arthur W., Ph.D. (Nov. 5, '04) Gen. Mgr. The Western Reserve Chem., Co., 3434 E. 93d St., Cleveland, Ohio.

BUTCHER, J. C. (Sept. 27, '16) Prop. J. C. Butcher Co., Hood River, Oregon.

BUSTOS, Enrique (Oct. 3, '17) Engineer, Baltimore Copper Works; mailing address, 1521 Mt. Vernon St., Philadelphia, Pa.

BUTTERS, Chas. (July 1, '05) 6272 Chabot Road, Oakland, Cal.

BUTTFIELD, W. J. (Oct. 23, '14) Pres. Vulcan Detinning Co., Sewaren, N. J.

BUTTS, Allison (Dec. 31, '14) Asst. Editor, Mineral Industry, Box 89, Bethlehem, Pa.

CALDER, A. R. (May 26, '10) Met., Bethlehem Steel Co., Steelton, Pa.; mailing address, 2200 Bellevue Road, Harrisburg, Pa.

CALHANE, D. F. (June 1, '15) Prof. Electro-Chemistry, Worcester Polytechnic Inst., Worcester, Mass.

CALLEN, A. S. (Apr. 24, '14) Res. Chemist, Wedge Mechanical Furnace, Philadelphia; mailing address, 3510 N. 16th St.

CAMERON, Frank K., Ph. D. (Oct. 7, '05) Consult. Chemist and Chem. Eng., 3207 19th St., N. W., Washington, D. C.

CAMERON, Walter S (Apr. 3, '02) 239 W. 136th St., New York City.

CAMPBELL, P. A. (May 2, '17) Res. Eng., 307 Verona Ave., Newark, N. J.

CANBY, Robt. C. (July 24, '14) Consulting Metallurgist, 334 So. Main St., Wallingford, Conn.

CANET, B. Charles (Aug. 26, '10) Gen. Mgr., 95 William St., New York City.

CANTLEY, Thomas (Mar. 27, '09) Gen. Mgr., Nova Scotia Steel & Coal Co., Ltd., New Glasgow, Nova Scotia, Canada.

CARDOEN, Remy (May 25, '17) P. O. Box 3463, Santiago de Chile.

CARGO, L. M. (May 2, '17) District Mgr., Westinghouse Elec. Mfg. Co.; mailing address, 1052 Gas & Elec. Bldg., Denver, Colo.

CARHART, Prof. H. S. (Apr. 3, '02) Prof. Emeritus of Physics, Univ. of Mich., Ann Arbor, Mich.; mailing address, 277 N. El Molino Ave., Pasadena, Cal.

CARLSON, Birger (Nov. 5, '04) Chemist, Electrochem. Work of Stockholm Superfosfat Aktiebolag of Stockholm, Sweden; res., Mansbo, Avesta, Sweden.

CARNEGIE, Ebenezer (Aug. 28, '14) Pres. and Gen. Mgr., The Electric Steel and Metals Co., Ltd., Welland, Ont., Canada.

CARRIER, C. F., Jr. (Mar. 5, '05) Dept. Mgr., Rollin Chem. Co.; mailing address, 1567-B Lee St., Charleston, W. Va.

CARRIER, S. C. (Sept. 27, '16) Sales Engr., Westinghouse Elec. & Mfg. Co., 165 Broadway, New York City; mailing address, 2042 Bedford St., Brooklyn, N. Y.

CARRIER, W. H. (Dec. 31, '15) Pres. and Chief Eng., Carrier Eng. Co.; mailing address, 603 Mutual Life Bldg., Buffalo, N. Y.

CARSE, David B. (Mar. 4, '05) President, David B. Carse & Co., 165 Broadway, New York City.

CARSON, F. L. (Aug. 25, '17) Gen. Mgr., Pacific Carbon Mfg. Co., 21st and Chanslor Ave., Richmond, Cal.

CARTER, F. E. (Aug. 25, '16) Physical Metallurgist, Baker & Co., Inc., care of Baker Platinum Works, Newark, N. J.

CARVETH, H. R., Ph.D. (Apr. 3, '02) Works Mgr., Niagara Electrochemical Co., Niagara Falls, N. Y.; res., 118 Buffalo Ave.

CARY, Charles R. (May 29, '09) Engineering Salesman, The Leeds & Northrup Co., 4901 Stenton Ave., Philadelphia, Pa.

CASE, Theodore W. (Dec. 30, '16) Auburn, N. Y.

CASE, Willard E. (Oct. 2, '02) 196 West Genesee St., Auburn, N. Y.

CASE, Willis W., Jr. (Nov. 27, '09) Denver Athletic Club, Denver, Colo.

CASSELBERRY, Harry (June 29, '07) 2218 4th Ave., Altoona, Pa.

CASTLE, S. N. (Jan. 27, '12) care of General Electric Co., 120 Broadway, New York City.

CATANI, Dr. Remo (Aug. 31, '07) E. E., 41, Via Babuino, Rome, Italy.

CAULKINS, E. B. (Jan. 26, '17) V. P. and Sec'y, Mich. Steel Castings Co., Detroit, Mich.

CHACE, R. T. (Feb. 24, '17) Salesman, Westinghouse Elec. and Mfg. Co.; mailing address, 228 4th St., Niagara Falls, N. Y.

CHADWICK, R. A., Jr. (July 27, '17) Asst. Melter, Anniston Steel Co., Anniston, Ala.

CHALAS, Adolphe (May 29, '09) care of Chalas & Sons, Finsbury Pavement House, Finsbury Pavement, London, E. C., England.

CHANDLER, Dr. C. F. (Jan. 8, '03) Prof. of Chem., Columbia Univ., New York.

CHANEY, N. K. (Mar. 27, '14) Res. Chemist, The National Carbon Co., Cleveland, Ohio; mailing address, 1614 Wyandotte Ave., Lakewood, Ohio.

CHAPIN, Dr. H. C. (Apr. 22, '15) Research Chemist, Nat. Carbon Co.; mailing address, 1523 Elbur Ave., Lakewood, Ohio.

CHAPPELL, Wm. C. (June 28, '12) Hydro-Electric Dept., Hobart, Tasmania.

CHEDSEY, Wm. R. (Apr. 24, '09) Assoc. Prof. of Mining, Penna. State College, State College, Pa.; mailing address, 607 West College Ave.

CHERRY, Louis B. (Dec. 31, '15) Electrochemist and Elec. Eng., care of C. & C. Development Co., Kansas City, Mo.

CHIARAVIGLIO, Dino (Apr. 3, '02) Piassa Esquilino 23, Rome, Italy.

CHILD, Hugh A. (Aug. 25, '16) Mgr. and Supt., Bayard Chem. Co., Woodbridge, N. J.

CHILLAS, Richard B., Jr. (May 5, '10) Chemical Engr., National Carbon Co., Cleveland, Ohio; res., 1276 W. 112th St.

CITO, C. C. (Sept. 26, '08) Consulting Engineer, Director and Gen. Mgr., of the Mines de Monte Zippiri Sardaigne, 10 Rue Henri Marichal, Brussels, Belgium.

CLAMER, G. H., (Apr. 3, '02) Ajax Metal Co., Frankford Ave. and Richmond St., Philadelphia, Pa.

CLAPP, E. H. (Sept. 4, '03) Vice-Pres., Penobscot Chem. Fibre Co., 49 Federal St., res., 490 Beacon St., Boston, Mass.
CLAPP, Joseph F. (Nov. 27, '09) Metallurgist, Rare Metals Corporation, Los Angeles, Cal.; res., 3118 Humboldt St., Los Angeles, Cal.
CLARK, Walter G. (Sept. 28, '07) Pres. Parker-Clark Electric Co., 135 Broadway, New York City.
CLARK, Wm. J. (Apr. 3, '02) Gen. Mgr. Foreign Dept., Gen. Elec. Co., 44 Broad St., New York City.
CLARK, Wm. W. (May 27, '11) Gen. Mgr., Noble Electric Steel Co., Heroult, Cal.
CLARKE, Eben B. (June 25, '09) 5201 Westminster Place, Pittsburgh, Pa.
CLARKE, F. E., Ph. D. (Apr. 3, '02) Prof. of Chem., Dept. of Chem., West Virginia Univ., Morgantown, W. Va.
CLEMENTS, Frank O. (Apr. 29, '11) Director, Dayton Research Labs. Co.; mailing address, 101 Far Hills Ave., Oakwood, Dayton, Ohio.
CLEVENGER, G. H. (Aug. 27, '09) Consulting Engineer, Butters Salvador Mines; mailing address, 381 Hawthorne Ave., Palo Alto, Cal.
CLYMER, W. R. (May 30, '08) Factory Mgr., National Carbon Co.; mailing address, 13985 Lake Ave., Cleveland, Ohio.
COATES, Jesse (May 29, '09) 40 Ocean Ave., Lynn, Mass.
COFFIN, F. P. (May 25, '12) Electrical Engineer, General Electric Co., Schenectady, N. Y.; res., 69 Bedford Road.
COGGESHALL, G. W. (Apr. 3, '02) Chem. Eng., The Inst. of Industrial Res., Washington, D. C.; mailing address, 2229 California St.
COHO, H. B. (Apr. 3, '02) United Lead Co., 111 Broadway, New York City.
COHOE, W. P. (Oct. 28, '09) Consulting Chemist, 111 Broadway, New York City.
COLBY, Ed. A. (Apr. 3, '02) Supt., Baker Platinum Works, Newark, N. J.
COLE, Chas. S. (Dec. 31, '15) Captain, Ordnance Dept., M. S. R.; mailing address, 820 Penobscot Bldg., Detroit, Mich.
COLE, Edward R. (June 25, '09) Superintendent Plants Nos. 1 and 2, Acheson Graphite Co., Niagara Falls, N. Y.
COLLETT, Emil (Nov. 24, '11) 9 Lokkeveien, Kristiana, Norway.
COLLETT, Ove (Feb. 23, '12) Met. Engr., Munkedamsveien 27, Kristiana, D. Norway.
COLLINS, Fred L. (Oct. 29, '08) Elec. Engr., Dominion Iron and Steel Co., 160 Whitney Ave., Sydney, Cape Breton, Nova Scotia, Canada.
COLVOCORESSES, Geo. M. (Dec. 31, '09) Gen. Mgr., Consolidated Arizona Smelting Co., Humboldt, Arizona.
COMEY, Arthur M. (Apr. 29, '11) Director, Eastern Laboratory, E. I. du Pont de Nemours Powder Co., Chester, Pa.; res., 424 E. 13th St.
COMSTOCK, Chas. W. (June 27, '13) Consult. Eng., 1235 First Nat. Bank Bldg., Denver, Colo.
COMSTOCK, D. F. (Mar. 27, '14) Eng. and Assoc. Prof. of Physics, Mass. Inst. of Technology, Cambridge, Mass.
COMSTOCK, R. L. (June 30, '17) Chem. Eng., Brown Co., Berlin, New Hampshire.
CONKLIN, E. B. (Oct. 21, '16) Eng. Semet Solvay Co., 236 W. Borden Ave., Syracuse, N. Y.
CONNELL, H. R. (May 26, '10) Allegheny Steel Co., Brackenridge, Pa.; res., Tarentum, Pa.
CONVERSE, Vernon G. (Oct. 29, '08) General Mgr. and Chief Engineer, The Ontario Power Co.; P. O. Box 496, Niagara Falls, N. Y.
CONVERSE, W. A. (Nov. 27, '09) Secretary and Chemical Director, Dearborn Drug and Chemical Co., Chicago, Ill.; mailing address, 4320 Greenwood Ave.
COOK, Edw. B. (May 5, '10) 1568 E. 108 St., Cleveland, Ohio.
COOK, Roy J. (June 30, '17) Student, Mass. Inst. of Tech.; mailing address, 940 Albernarle Road, Brooklyn, N. Y.
COOLIDGE, Wm. D. (June 27, '13) Asst. Director, Res. Lab., Gen. Elec. Co., Schenectady, N. Y.
COOPER, H. C. (Nov. 26, '15) Prof. of Physical Chem., Syracuse University, Syracuse, N. Y.
COOPER, K. F. (Feb. 27, '09) Vice-Pres., American Cyanamid Co., 511 Fifth Ave., New York City.
COPE, F. T. (Jan. 25, '13) Enginecr, care of Electric Furnace Co., Alliance, Ohio.
COPEMAN, L. G. (Aug. 25, '16) Designing Eng., Copeman Electric Stove Co., 1715 Detroit St., Flint, Mich.
CORBIN, J. Ross (May 26, '10) Boyerstown, Pa.
CORNING, Christopher R. (July 21, '11) 36 Wall St., New York City; res., Tuxedo Park, N. Y.
CORNTHWAITE, Hayden (Sept. 25, '09) care of 2 The Avenue, Brimsdown, Middlesex, England.
CORSE, Wm. M. (May 5, '10) Mgr. Bronze Dept., The Titanium Bronze Co., Inc., Niagara Falls, N. Y.; res., 106 Morris Ave., Buffalo, N. Y.
COTTRELL, F. G. (Dec. 28, '12) Chief Metallurgist, U. S. Bureau of Mines, Washington, D. C.

COWAN, Wm. A. (May 5, '10) Assistant Chemist, Research Laboratories, National Lead Co., 129 York St., Brooklyn, N. Y.

COWLES, Alfred H. (Apr. 3, '02) The Elec. Smelt. and Aluminum Co., P. O. Box 84, Sewaren, N. J.

COWLES, Harry D. (Dec. 28, '12) Res. Chemist, 166 N. 17th St., East Orange, N. J.

COWPER-COLES, S. (Oct. 10, '03) 1 and 2 Old Pye St., Westminster, London, S. W., England; mailing address, "The Cottage," French St., Sunbury on Thames.

COWPERTHWAIT, Arthur D. (Aug. 25, '16) Chem. Eng., The Edmunds & Jones Corp., Detroit, Mich.

COX, G. E. (Apr. 3, '02) Works Mgr., American Cyanamid Co., Niagara Falls, N. Y.,

COX, Harold N. (Oct. 21, '16) Director, Edison Central Research Lab., Silver Lake, New Jersey.

COX, Samuel F. (June 1, '15) 1st Lieut.; mailing address, care of Mr. E. H. Hanna, Springdale, Pa.

CRABTREE, Prof. Fred. (May 29, '09) Prof. of Metallurgy and Mining, Carnegie Technical Schools, Pittsburgh, Pa.; mailing address, 6214 Stewart St.

CRAFTS, Walter N. (Aug. 25, '11) President, Crucible Steel Forge Co., Cleveland, Ohio; res., Oberlin, Ohio.

CRANE, F. D. (Oct. 29, '08) Research Chemist, Synfleur Sci. Labs., Monticello, N. Y.; mailing address, 28 Hillside Ave., Montclair, N. J.

CRANSTON, John (Oct. 21, '16) Chemist, Penna. Salt Mfg. Co.; mailing address, 725 North Biddle Ave., Wyandotte, Mich.

CRAWFORD, C. A. (July 27, '17) Res. Asso., American Sheet and Tin Plate Co.; mailing address, 6709 McPherson Blvd., Pittsburgh, Pa.

CREIGHTON, Elmer E. F. (Apr. 3, '02) Consult. Eng., General Electric Co., Schenectady, N. Y.; mailing address, 27 Wendell Ave.

CREIGHTON, H. J. (June 2, '16) Asst. Prof. of Chemistry, Swarthmore College, Swarthmore, Pa.

CRIDDLE, E. B. (Apr. 26, '17) Gen. Agent, Southern Sierras Power Co., Riverside, Cal.

CRIDER, J. S. (May 9, '03) Secretary, National Carbon Co., Cleveland, Ohio.

CROCKER, Jas. R. (Feb. 25, '11) Chief Eng., Permutit Co., 30 E. 42d St., New York City; res., 202 W. 79th St., New York City.

CROSBY, E. L. (Dec. 28, '12) Sales Engr., 18 Washington Blvd., Detroit, Mich.

CUMMINGS, William J., C.E. (Dec. 31, '09) Chief Engineer, E. I. du Pont Powder Co., Haskell, N. J.

CUNNINGHAM, Frederick Wm. (Dec. 29, '11) Advisory Consultant, care of Powdered Coal Eng. and Equipment Co., 2401 Washington Blvd., Chicago, Ill.; mailing address, 821 Lake St., Oak Park, Ill.

CURFMAN, F. G. (July 25, '15) 519 International Bldg., St. Louis, Mo.

CURTISS, John L. (Mar. 26, '15) Local Eng., Buffalo, N. Y.; mailing address, Gen. Elec. Co., 10th floor, Gen. Elec. Bldg., Buffalo, N. Y.

CUSHING, H. M. (May 27, '14) Engineer, Buffalo General Elec. Co., Buffalo, N. Y.; mailing address, 1949 Hertel Ave.

CUSHMAN, O. E. (July 27, '17) Res. Chem., 2333 Channing Way, Berkeley, Cal.

CUTTS, V. O. (June 27, '13) Electrometallurgist, Town Hall Chambers, 87 Fargate, Sheffield, England.

CZARNECHI, F. C. (July 25, '15) 31 Phelps Road, Upper Ridgewood, N. J.

DABOLT, Newton E. (Apr. 26, '13) Sales Mgr., 185 Church St., New Haven, Conn.; mailing address, 190-192 Willard St.

DAFT, Leo (Mar. 27, '09) Consulting Electrical Engineer, Rutherford, N. J.

DALBEY, G. E. (Sept. 27, '16) care of Benj. Harris Metals Co., Chicago Heights, Ill.

DAINES, W. P. (Sept. 27, '16) Chief Chemist, Columbus Iron & Steel Co., Columbus, Ohio.

DALMEIDA, J. A. (Dec. 30, '16) Chief Clerk, Electrolytic Tank House, Chile Exploration Co., Chuquicamata, Chile.

DALTON, A. C. (Mar. 24, '16) care of Robt. Hyde & Son Ltd., Abbeydale Foundry, Woodseats, Sheffield, England.

DANTSIZEN, Christian (Apr. 26, '17) Chem., Res. Lab., Gen. Elec. Co., Schenectady, N. Y.

DAPPLES, Alfred (Oct. 29, '08) Directeur, Societe Fabbricaziane del Aluminio, Bussi-Afficienne, Aguila, Italy.

DARLINGTON, H. T. (Oct. 26, '17) Met. Eng., Penna. Salt Mfg. Co., Natrona, Pa.

DARRAH, Wm. A. (Aug. 25, '16) Chief Eng., Ohio Brass Co., Mansfield, Ohio.

DARRIN, Marc (Nov. 23, '17) Res. Chem., H. Koppers Co., Mellon Inst., Pittsburgh, Pa.

DAVIDSON, T. R. (Sept. 27, '16) Director, The Tos. Davidson Mfg. Co., Ltd., P. O. Box 700, Montreal, Canada.

DAVIS, D. L. (Aug. 7, '02) Supt. The Salem Elec. L. and P. Co.; res., 299 Lincoln Ave., Salem, Ohio.

DAVIS, F. W. (May 24, '13) Fuel Chemist, Alan Wood Iron and Steel Co., Conshohocken, Pa.

DAVIS, Hugh N. (Sept. 27, '16) Supt., Keokuk Electro-Metals Co.; mailing address, 512 N. 10th St., Keokuk, Iowa.
DAVIS, R. W., Jr. (Feb. 27, '09) Cons. Eng., 305 Highland Ave., Jenkintown, Pa.
DAVISON, A. W. (Mar. 24, '16) Charge of Phy. and Applied Electrochem., Dept. of Chemistry, Univ. of Cincinnati, Ohio.
DEACON, Ralph W. (May 5, '10) Supt., United States Metals Ref. Co., Chrome, New Jersey.
DEAGLE, L. M. (Oct. 21, '16) Electrician, Cataract Elec. Co. Ltd., Orangeville, Ont., Canada.
DE BEERS, F. M. (May 29, '09) Pres. & Gen. Mgr., Swenson Evaporator Co., 945 Monadnock Bldg., Chicago, Ill.
DE GEOFROY, Antoine (Jan. 25, '13) 12 rue du Bouquet, de Long Champs, Paris, France.
DE JOANNIS, Harry (May 22, '14) Managing Editor, "Brass World & Platers' Guide," Bridgeport, Conn.
DE MEDEIROS, Trajano (Jan. 29, '10) Rua de Sao Jose, No. 76, Rio De Janeiro, Brazil.
DE MILES, Paul (Aug. 25, '17) 167 West 18th St., New York City.
DEMOREST, D. J. (Aug. 25, '16) Prof. of Met., Dept. of Metallurgy, Ohio State Univ., Columbus, Ohio.
DE NEUFVILLE, Dr. R. (Feb. 5, '03) Junghofstrasse 14, Frankfort a/M, Germany.
DEPPE, Wm. P. (Dec. 30, '16) Pres. Deppe Motors Corp., 127 Duane St., New York City.
DESHLER, Geo. O. (Oct. 24, '13) Supt., Mines Operating Co., Butte, Mont.
DE SOUZA, Edgard (Dec. 31, '09) Chief Electrical Engineer, The S. Paulo Tramway, Light and Power Co., Ltd., S. Paulo, Brazil; mailing address, Caixa 162,
DETWILER, J. G. (Dec. 30, '16) 1st Lieut., Co. "H," 347th Infantry, Camp Pike, Arkansas.
DEVEREUX, Washington (Jan. 6, '06) Inspector, Philadelphia Fire Underwriters' Association, 1625 N. 29th St., Philadelphia, Pa.
DEVERS, Philip K., Jr. (Jan. 27, '12) Research Engineer, 254 W. York Ave., York, Pa.
DEWEY, Bradley (Jan. 28, '11) Director, Research Laboratory, Am. Sheet and Tin Plate Co., 210 Semple St., Oakland, Pittsburgh, Pa.
DEWEY, Edwin S. (Dec. 26, '13) 162 Elmwood Ave., Newark, N. J.
DEWEY, F. P. (Apr. 2, '04) Assayer, Mint Bureau, Treas. Dept., Washington, D. C.
DIAZ-OSSA, Prof. Belisario (June 25, '09) Prof. of Technology, University of Chile, Casilla No. 962, Santiago, Chile, South America.
DIETRICHSON, J. G. (Dec. 30, '17) Instructor in Chem., Univ. of Illinois, Chem. Bldg., Urbana, Ill.
DITTMAR, Carl (June 2, '16) 2212 Union Central Bldg., Cincinnati, Ohio.
DIXON, Jos. L. (Oct. 23, '14) Engineer, John A. Crowley Co., 120 Liberty St., New York City.
DODGE, Col. David C. (Apr. 29, '11) 1654 Broadway, Denver, Colo.
DODGE, W. E. (Feb. 24, '17) Chemist, 415 W. Highland Ave., Shawnee, Okla.
DODSON, Fred W. (July 27, '17) Student, Mass. Inst. of Tech.; mailing address, 1319 N St., N. W., Washington, D. C.
DOERFLINGER, Wm. F. (July 3, '02) Perry Austin Mfg. Co., Grasmere, Staten Island, New York City.
DOERSCHUK, Victor C. (Apr. 29, '11) Massena, N. Y.
DONY-HENAULT, Prof. O. (Feb. 27, '14) 40, Avenue de Bertaimont, Al Ecole des Mines, Mons, Belgium.
DOOLITTLE, C. E. (May 9, '03) Consulting Hyd. & Elec. Engr., V.-Pres. and Gen. Mgr., The Roaring Fork Elec. Light and Power Co., Aspen, Col.
DOREMUS, Dr. Chas. A. (Apr. 3, '02) 55 W. 53d St., New York City.
D'ORNELLAS, Thomas V. (Nov. 21, '08) Cons. Eng., 22 Bajada Balta, Miraflores, Lima, Peru, S. A.
DORR, J. V. N. (Nov. 28, '13) Pres., The Dorr Co., 17 Battery Place, New York City.
DORSEY, H. G. (Oct. 3, '12) Gloucester, Mass.
DOTY, Ernest L. (Sept. 26, '08) Dist. Eng., Westinghouse El. & Mfg. Co., 6 and 8 Lock St., Buffalo, N. Y.; res., 546 Potomac Ave.
DOUBLEDAY, Ralph S. (Aug. 25, '16) Chief Chemist, Marden Orth & Hastings Co.; mailing address, 564 Park Ave., East Orange, N. J.
DOUGHERTY, John W. (Feb. 27, '09) President, Pittsburgh Crucible Steel Co., Pittsburgh, Pa.
DOUGLAS, J. (Nov. 27, '09) The Phelps-Dodge Co., 99 John St., New York City.
DOW, Herbert H. (Apr. 3, '02) Midland, Mich.
DOWNES, Arthur C. (July 30, '09) Chemist, National Carbon Co., Fostoria, Ohio.
DRAEGER, A. Bernhard (Nov. 26, '07) Draeger Werke, Lubeck, Germany.
DRAKE, Bryant S. (Feb. 24, '17) Chem. Eng., Union Iron Wks., San Francisco, Cal.; mailing address, 3850 Colby St., Oakland, Cal.
DREFAHL, L. C. (Oct. 22, '15) Res. Chem., Grasselli Chemical Co., 3132 W. 15th St., Cleveland, Ohio.

DREYFUS, Dr. Wm. (Dec. 4, '02) Chem. and Mgr., West Disinfectant Co., 57 E. 96th St., New York City.
DRINKER, Philip H. (Jan. 26, '17) 2d Lieut., U. S. Aviation Corps, Research Dept., care of Chief of Aviation, A. E. F., France.
DRIVER, Wilbur B. (Aug. 25, '16) Vice Pres., Driver-Harris Co., Newark, N. J.
DROBEGG, Dr. Gustave (Jan. 8, '03) Supt. Plant D, Butterworth-Judson Corp., P. O. Box 273, Newark, N. J.
DRYER, Ervin (Sept. 4, '03) Peoples Gas Bldg., Chicago, Ill.
DUDLEY, Boyd, Jr. (Nov. 28, '13) Asst. Prof. of Metallurgy, Penna. State College, State College, Pa.
DuFAUR, J. B. (June 1, '07) care of G. DuFaur, Turramurra, Sydney, N. S. W., Australia.
DUNCAN, Thos. (Nov. 6, '03) Vice-Pres. and Gen. Mgr., Duncan Elec. Mfg. Co., 312 S. 6th St., LaFayette, Ind.
DUNLAP, Orrin E. (July 31, '08) Sec'y, Int. Acheson Graphite Co., Niagara Falls, N. Y.
DUNN, J. J. (Feb. 25, '11) General Supt. and Chief Engr., Shelby Steel Tube Co., Elwood City, Pa.
DUNNINGTON, Prof. F. P. (Apr. 6, '11) Professor of Analytical and Industrial Chemistry, University of Virginia, University, Va.
DU PONT, Irenee (Apr. 24, '09) V. Pres., E. I. du Pont de Nemours Powder Co., Wilmington, Del.
DU PONT, Pierre S. (Jan. 29, '09) Treas., E. I. du Pont de Nemours Powder Co., Wilmington, Del.
DUSCHAK, L. H. (June 25, '09) Chem. Eng., U. S. Bureau of Mines, Mining Bldg., Berkeley, Cal.; mailing address, 1301 Tamalpais St.
DUSHMAN, Saul (June 25, '09) Res. Lab., General Elec. Co., Schenectady, N. Y.
DUURLOO, F. (Oct. 21, '16) Asst. Sec'y, W. R. Grace & Co., N. Y.; mailing address, 131 Caryl Ave., Yonkers, N. Y.
DUVAL, Alexander L. (Oct. 26, '17) Chief Chem., Hazel Atlas Glass Co.; mailing address, 315 E. Beau St., Washington, Pa.
DWIGHT, Arthur S. (Feb. 26, '10) Consulting, Mining and Met. Eng., 29 Broadway, New York City.
DYRSSEN, W. (Jan. 23, '14) Metallurgist, U. S. Steel Corp., 71 Broadway, New York City.
EAGLE, Henry Y. (Nov. 30, '12) Room 1227, 42 Broadway, New York City.
EASTMAN, Herbert C. (May 5, '10) Manager and Owner, The Ontario-Colorado Gold Mining Co., and The Colo.-Wyoming Power & Irrigation Co., 719-720 Equitable Bldg., Denver, Col.
EASTMAN, H. M. (Feb. 25, '16) Supt., The National Radium Inst.; mailing address, 739 S. Washington St., Denver, Colo.
EATON, I. C. (June 30, '17) Union Carbide Co. of Canada, Welland, Ont., Canada.
EBERWEIN, Samuel J. (May 25, '17) 1049 Main St., Slatington, Pa.
ECKER, Howard, Jr. (Oct. 21, '16) 3115 Woodburn Ave., Cincinnati, Ohio.
EDE, Joseph A. (Oct. 29, '10) Consulting Mining Eng., Illinois Zinc Co., La Salle, Illinois.
EDISON, Thos. A. (Apr. 4; '03) Orange, N. J.
EDMANDS, I. R. (Aug. 7, '02) Consult. Eng., Union Carbide Co., Niagara Falls, N. Y.; mailing address, 167 Buffalo Ave.
EDSTROM, J. Sigfrid (Nov. 5, '04) Managing Dir., General Electric Co. of Sweden, Vesteras, Sweden.
EDWARDS, John B., Jr. (May 2, '17) Chem. Eng., care of Illinois Zinc Co., Peru, Illinois.
EGLIN, Wm. C. L. (July 1, '04) Elec. Eng., 1000 Chestnut St., Philadelphia, Pa.
EIMER, A. (Dec. 4, '02) Eimer & Amend, 205-211 Third Ave., New York City.
EKELEY, John B. (Feb. 21, '13) Prof. of Chemistry, Univ. of Colo., Boulder, Colo.; res., 525 Highland Ave.
ELDRIDGE, Samuel E. (Sept. 27, '15) Special Agent, The American Rolling Mill Co., Middletown, Ohio.
ELLIOTT, Geo. K. (Dec. 30, '16) Chief Chem. and Met., The Lunkenheimer Co., Cincinnati, Ohio.
ELLIS, Carleton (Jan. 23, '14) Industrial Res. Chemist, 92 Greenwood Ave., Montclair, N. J.
EMANUEL, Louis V. (Oct. 17, '07) care of River Smelt. & Ref. Co., Florence, Colo.
EMERSON, Harrington (Feb. 25, '11) President, The Emerson Co., 30 Church St., New York City.
EMERY, A. L. (Apr. 3, '02) Smith, Emery & Co., Chem. and Met. Engs., Howard and Hawthorne Sts., San Francisco, Cal.
EMERY, W. L. (Sept. 26, '08) Foreman of Meter Dept., Utah Lt. & Ry. Co.; mailing address, 43 N. 7th St., Salt Lake City, Utah.
ENGELHARD, Chas. (May 29, '09) President, American Platinum Works, Newark, N. J.; mailing address, Hudson Terminal Bldg., 30 Church St., New York City.

ENGELHARDT, Victor (Dec. 4, '02) Siemens & Halske Co.; res., Charlottenburg, Oranienstrasse 18, Germany.
ERHART, W. H. (Dec. 27, '07) 11 Bartlett St., Brooklyn, N. Y.
ERICSON, Eric J. (Oct. 24, '13) 5045 Cullom Ave., Chicago, Ill.
EURICH, E. F. (Nov. 27, '09) Mining and Metallurgical Engineer, 15 William St., New York City; res., Montclair, N. J.
EUSTIS, Augustus H. (Dec. 31, '09) Mining Engineer, 131 State St., Boston, Mass.
EVANS, H. S. (Apr. 3, '02) Prof. Elec. Eng., Univ. of Col., Boulder, Colo.
EVERETTE, Dr. Willis E. (July 30, '09) Consulting Chemical and Mining Engineer, Tacoma, Wash.
FALTER, Philip H. (Aug. 25, '11) Gen. Mgr., Shawinigan Electro Products Co., Baltimore, Md.; mailing address, 1606 Lexington St. Bldg.
FARNHAM, Frederick F. (May 26, '10) American Bridge Co., Box 55, Ambridge, Penna.
FARROW, Percival R. (June 30, '16) Power Station Supt., Kaministiquia Power Co., Ltd., Kakabeka Falls, Ontario, Canada.
FARUP, Dr. P. (Jan. 28, '16) Tekniske Hoiskole, Trondhjem, Norway.
FATTINGER, Dr. Franz (Mar. 23, '12) Treibach, Carinthia, Austria.
FAWCETT, Lewis H. (May 25, '12) Chemist, care of High Speed Tools, Corp., 16 Broadway, Toledo, Ohio.
FAWCETT, Percy (Sept. 25, '09) Director, Thomas Firth & Sons, Ltd., Sheffield, England.
FEHNEL, J. Wm. (June 26, '14) 703 N. Main St., Bethlehem, Pa.
FERNBERGER, H. M. (Jan. 8, '04) 66 Millington Ave., Newark, N. J.
FERNEKES, Gustave (Oct. 22, '15) Chemist, The Am. Conduit Mfg. Co., Oakmont, Pa.
FERRY, Chas. (June 21, '11) Metallurgist, Bridgeport Brass Co., Bridgeport, Conn.
FICHTER-BERNOULLI, Prof. Dr. F. (Nov. 26, '07) 35 Neubadstrasse, Basel, Switzerland.
FINCKH, Dr. Carl (Apr. 24, '14) Asst. Director, Deutsche Gasglühlicht Aktiengesellschaft (Auergesellschaft) Rotherstrasse 8/12, Berlin 0.17, Germany.
FINK, Dr. Colin G. (Nov. 26, '07) Head of Labs., Chile Exploration Co., 202d St. and 10th Ave., New York City.
FINNEY, John H. (Oct. 22, '15) Mgr. Southern Office, Aluminum Co. of America, 509 Met. Bank Bldg., Washington, D. C.
FISCHER, Siegfried (Feb. 25, '11) Golden, Colo.
FISH, Job, Jr. (Jan. 28, '16) Works Mgr., The Otis Elevator Co.; mailing address, Box 156, Buffalo, N. Y.
FISHER, Henry W. (May 26, '10) Chief Engineer, Standard Underground Cable Co., Perth Amboy, N. J.
FITZGERALD, F. A. J. (Apr. 3, '02) The FitzGerald Laboratories, Inc., Highland and Vermont Aves., Niagara Falls, N. Y.
FITZ GIBBON, R. (Apr. 3, '02) Room 212, 43 Leonard St., New York City.
FLAGG, Frederick P. (Oct. 26, '17) Chief Chem., Waltham Watch Co., Waltham, Mass.
FLANNERY, Jas. J. (May 26, '10) President, American Vanadium Co., 325 Frick Bldg., Pittsburgh, Pa.
FLASHMAN, H. W. (Feb. 24, '17) The Foundation Co., 233 Broadway, New York City.
FLEMING, R. (Apr. 3, '02) Box 72, Swampscott, Mass.
FLEMING, S. H. (Nov. 26, '10) Research Laboratory, National Carbon Co., Cleveland, Ohio.
FLETCHER, W. E. (Aug. 25, '16) Chem. Director, Atlas Powder Co., Wilmington, Delaware.
FLIESS, R. A. (Sept. 4, '02) 99 Claremont Ave., New York City.
FLINN, Elmer H. (July 27, '17) Student, Lehigh Univ., Bethlehem, Pa.; mailing address, 334 E. Broad St.
FOERSTERLING, Dr. Hans (Apr. 3, '02) care of The Abor Farm, Jamesburg, N. J.
FOGARTY, John A. (Oct. 21, '16) Asst. Supt., Electrolytic Plant, Brown Co.; mailing address, 166 Prospect St., Berlin, N. H.
FOLEY, Chas. B. (July 25, '13) 170 Broadway, New York City.
FOOTE, Arthur DeWint (Feb. 27, '09) Superintendent, North Star Mines, Grass Valley, Cal.
FORBES, Geo. S. (May 26, '15) Asst. Prof. of Chem., Harvard University; mailing address, Boylston Hall, Cambridge, Mass.
FORCHHEIMER, Dr. Phil. J. (Dec. 26, '13) Tech. Director, Gesellschaft Electrometallurgie, 20 Bucherstrasse, Nurnberg, Germany.
FORSSELL, Dr. J. (June 1, '07) 70 A Karlbergsvaegen, Stockholm, Sweden.
FOSTER, Chas. L. (June 30, '16) Sales Mgr., The Electric Furnace Co., 505 Alliance Bank Bldg., Alliance, Ohio.
FOSTER, Oscar R. (Apr. 30, '08) Chemistry Teacher, 203 Eighth Ave., Brooklyn, New York.

FOSTER, Raimond D. (June 1, '15) Treas., care of The Hanson and Van Winkle Co., 269 Oliver St., Newark, N. J.

FOUST, Thos. B. (Jan. 29, '09) care of Bon Air Coal & Iron Corp., Allens Creek, Tenn.

FOWLER, Edw. J. (July 27, '17) Mgr., care of Pacific Foundry Co., 18th and Harrison Sts., San Francisco, Cal.

FOWLER, R. E. (Nov. 6, '03) Chemist, The National Electrolytic Co., Niagara Falls, N. Y.

FOWLER, Samuel S. (Apr. 3, '02) Min. Eng., P. O. Drawer 1024, Nelson, B. C., Canada.

FOX, Herbert W. (Oct. 26, '17) Chem. Eng., The Dorr Co.; mailing address, P. O. Box 204, Westport, Conn.

FOX, William J. (Dec. 28, '17) Gen. Mgr., Mohawk Improvement Co., Fort Plain, N. Y.

FRALEY, Jos. C. (Apr. 3, '02) Attorney-at-Law, 1815 Land Title Bldg.; res., 1833 Pine St., Philadelphia, Pa.

FRANK, John J. (Dec. 31, '15) Asst. Transformer Eng., General Electric Co.; mailing address, 43 George St., Pittsfield, Mass.

FRANK, Karl G. (Feb. 27, '09) Representative, Siemens & Halske, A. G., West St. Bldg. (90 West St.) Room 408, New York City.

FRANKFORTER, G. B. (Apr. 3, '02) Prof., Univ. of Minnesota, Minneapolis, Minn.

FRANKLIN, Milton W. (May 29, '09) Engineer, Ozonator Dept., Gen. Elec. Co., Bloomfield, N. J.; mailing address, 250 West Somerset St., Philadelphia, Pa.

FRARY, Francis C. (Aug. 31, '07) Res. Chemist, care of Aluminum Co. of America, mailing address, 14 C St., Niagara Falls, N. Y.

FREAS, Thos. B. (Oct. 29, '10) Havemeyer Hall, Columbia University, New York City.

FREDERICK, Geo. E., Jr. (Jan. 29, '09) 169 Columbia Heights, Brooklyn, N. Y.

FREEMAN, Gay N. (July 30, '09) Assayer and Analytical Chemist, Thermopolis, Wyoming.

FREEMAN, John R., Jr. (Oct. 26, '17) Lab. Asst., Bureau of Standards; mailing address, 1923 Calvert St., Washington, D. C.

FREUD, B. B. (Jan. 28, '16) Assoc. Prof. of Chem., Armour Inst. of Tech., Chicago, Ill.

FRICK, Frederick F. (Feb. 26, '15) Res. Eng., Anaconda Copper Min. Co., Anaconda, Mont.

FRICKEY, Royal E. (Sept. 20, '11) Eng., 505 S. Jefferson St., Chicago, Ill.

FRIES, Harold H., Ph.D. (May 1, '06) 92 Reade St., New York City.

FULLER, G. Prescott (Dec. 30, '16) Chief Chemist, National Electrolytic Co., Niagara Falls, N. Y.

FURNESS, Radclyffe (May 29, '09) Asst. Supt., Midvale Steel and Ordnance Co., Philadelphia, Pa.

GABRIEL, Geo. A. (Apr. 3, '02) 165 Marrett St., Cumberland Mills, Me.

GABY, Frederick A. (Mar. 26, 1915) Chief Eng., Hydro-Elec. Power Commission of Ontario; mailing address, 709 Continental Life Bldg., Toronto, Can.

GAHL, Dr. Rudolf (June 6, '03) care of Inspiration Cons. Copper Co., Miami, Ariz.

GAILLARD, Lieut. David St. P. (Aug. 25, '16) 1st Lieut., Ordnance Officers' Reserve Corps, U. S. A.; mailing address, Division T, Office of Chief of Ordnance, Washington, D. C.

GAINES, Richard H. (Oct. 17, '07) Chemist, New York Board of Water Supply, 49 Lafayette St., New York City; res., 35 W. 38th St.

GALL, Henry (Apr. 2, '04) Societe de Electrochimie, 2 Rue Blanche, Paris, France.

GANDILLON, Ami (Jan. 8, '04) Case 6219, Bourg de Four, Geneva, Switzerland.

GARDNER, Henry A. (Oct. 28, '09) The Institute of Industrial Research, Washington, D. C.

GARRATT, Frank (Feb. 21, '13) Latrobe Electric Steel Co., Latrobe, Pa.; mailing address, care of Mrs. Frank Garratt, 724 Weldon St.

GAYLORD, C. H. (June 30, '17) Salesman, Metal Locker Co.; mailing address, P. O. Box 800, Chicago, Ill.

GEESON, Arthur (Oct. 21, '16) Supt. Construction and Operation, Molybdenum Products Co., R. F. D. No. 1, Box 56 E, Tucson, Arizona.

GEGENHEIMER, R. E. (Feb. 27, '14) Chief Chemist, Mathiesen Alkali Works, Niagara Falls, N. Y.

GELSTHARP, Frederick (Aug. 25, '11) Chief Chemist, Pittsburgh Plate Glass Co., Creighton, Pa.; res., Tarentum, Pa.

GEPP, Herbert W. (Aug. 26, '10) General Manager, Amalgamated Zinc (De Bavays) Ltd., Broken Hill, N. S. W., Australia.

GERRY, M. H., Jr. (Apr. 3, '02) Gen. Mgr. and Chief Eng., Missouri River Power Co., Helena, Mont.

GERSTLE, John (May 27, '14) 592 W. 3d St., Dayton, Ohio.

GETMAN, F. H. (Apr. 24, '14) Asso. Prof. of Chemistry, Hillside Laboratory, Glenbrook Road, Stamford, Conn.

GEYLER, F. O. (Nov. 25, '15) Sec'y and Treas., Niagara Alkali Co., Niagara Falls, N. Y.
GIBBS, Arthur E. (Oct. 2, '02) Mfg. Chemist, care of Penna. Salt Mfg. Co., Widener Bldg., Philadelphia, Pa.
GIBSON, C. B. (May 26, '10) Sales Dept., Westinghouse Electric & Manufacturing Co., East Pittsburgh, Pa.; mailing address, 1656 Denniston Ave., Pittsburgh, Pa.
GIERTSEN, Sigurd (Nov. 24, '11) North Western Cyanamide Co., Odda, Norway.
GIFFORD, A. McK. (Feb. 23, '12) Eng. of Materials, General Electric Co., Pittsfield, Mass.; mailing address, 65 William St.
GIFFORD, Wm. E. (Apr. 3, '02) Baker & Co., 54 Austin St., Newark, N. J.
GILBERT, Harvey N. (Dec. 31, '15) Res. Chemist, Niagara Electrochemical Co., 442 10th St., Niagara Falls, N. Y.
GILBERTSON, H. A. (Apr. 2, '13) Mgr. and Jr. Member of Firm, Gilbertson & Son, 7561 Pratt Ave., Edison Park, Ill.
GILCHRIST, Peter S. (Apr. 3, '02) Chem. Eng., Charlotte, N. C.
GILLETT, Horace W. (Mar. 26, '10) U. S. Bureau of Mines, Morse Hall, Ithaca, N. Y.
GILLIES, P. McP. (Dec. 28, '12) Metallurgist, care of Hydro Electric Power and Metallurgical Co., Lombard Bldgs., 17 Queen St., Melbourne, Australia.
GILLIGAN, Frank P. (Jan. 23, '14) Treasurer, The Henry Souther Engineering Co., 11 Laurel St., Hartford, Conn.
GILLINGHAM, C. A. (Oct. 21, '16) Res. Chem., National Carbon Co.; mailing address, 1260 Edwards Ave., Lakewood, Ohio.
GIOLITTI, Federico, Ph.D. (Oct. 17, '07) Director General Steel Works, Ansaldo & Co., Cornigliano Ligure, near Genoa, Italy; mailing address, Regio Politecnics, Via dell Ospedale, Torino, Italy.
GIVEN, Guy C. (Oct. 3, '17) Chem., Atlas Powder Co.; mailing address, 238 E. Broad St., Tamaqua, Pa.
GLADSON, Prof. W. N. (Apr. 3, '02) Vice-Pres., University of Arkansas; mailing address, 820 W. Maple St., Fayetteville, Ark.
GLAZE, John B. (June 27, '13) Norton Co., Niagara Falls, N. Y.
GLENN, Earl R. (Oct. 26, '17) Teacher of Physical Science, The Lincoln School of Teachers College, Columbia Univ., 646 Park Ave., New York City.
GLENNIE, Robert D. (June 2, '16) Salesman, General Electric Co., Gluck Bldg., Niagara Falls, N. Y.
GOEPEL, Carl P. (Nov. 4, '05) Patent-counsel, 165 Broadway, New York City.
GOLDSCHMIDT, Dr. Hans (Nov. 6, '03) Chem. Mfr., Essen-Ruhr, Germany.
GOLDSCHMIDT, Heinrich (Oct. 27, '11) Kristiania, Drammensveien 82, Kristiania, Norway.
GOLDSMITH, N. J. (July 27, '17) Res. Chem., Beckman and Linden Eng. Corp.: mailing address, 860 Sutter St., San Francisco, Cal.
GOLDSTEIN, Philip (Mar. 26, '15) Prin., The Clerkenwell Plating Works, 155-7-9 Roseberg Ave., London, E. C., England.
GOODALE, Stephen L., A.M., E.M., (May 26, '10) Prof. of Metallurgy, University of Pittsburgh, School of Mines, Pittsburgh, Pa.
GOODRICH, Robt. R. (Jan. 23, '14) Prof. of Met., University of Idaho, Moscow, Idaho.
GOODSPEED, George M. (May 26, '10) Metallurgist, National Wks., National Tube Co., McKeesport, Pa.
GOODWIN, H. M., Ph.D. (Apr. 3, '02) Mass. Inst. of Tech., Cambridge, Mass.
GOODWIN, Joseph H. (Jan. 6, '06) Asst. Supt., National Carbon Co.; mailing address, 1501 McPherson Ave., Fremont, Ohio.
GOODWIN, Dr. Leo Frank (Oct. 29, '08) Chemistry Dept., Queen's University, Kingston, Ontario, Canada.
GOODWIN, W. L., D.Sc. (Apr. 3, '02) Director, School of Mining, Kingston, Ont., Canada.
GORDON, Prof. C. McC. (Apr. 3, '02) Lafayette College, Easton, Pa.
GOSROW, R. C. (Sept. 27, '16) Electrometallurgist, Blackstone Apts., 203 Juneau Ave., Milwaukee, Wis.
GOTOH, Issaku (May 2, '17) Metal Mining Dept. of Mitsubishi Goshikaisha, Tokyo, Japan.
GOULD, David F. (Aug. 25, '16) Chem., Res. Dept., Mexican Petroleum Corp., Destrehan, La.
GRAVELY, Julian S. (May 25, '12) 66 Elmwood Road, New Haven, Conn.
GRAVES, Walter G. (Mar. 5, '03) Supt., Graselli Chem. Co.; res., 1950 E. 90th St., Cleveland, Ohio.
GRAY, Jas. H. (Apr. 6, '11) Metallurgical Engr., U. S. Steel Corporation, 71 Broadway, New York City.
GREEN, H. M. (Oct. 23, '14) Supt. of Tankhouse, U. S. Metal Ref. Co., Chrome, N. J.; mailing address, Box 68.
GREEN, S. Stuart (Nov. 24, '16) Supt. Toledo Elec. Steel Organization, Box 67, Station G, Toledo, Ohio.
GREENLEE, Wm. B. (Nov. 26, '10) Secretary, Greenlee Foundry Co., Chicago, Ill.; res., 855 Buena Ave.

GREENWOOD, H. D. (Oct. 23, '14) Chief Chemist, care of U. S. Metals Ref. Co., Chrome, N. J.; res., 883 Colonia Road, Elizabeth, N. J.

GRIFFIN, Geo. B. (Feb. 24, '17) Mgr. Automobile Equipment Dept., Westinghouse Elec. & Mfg. Co.; mailing address, 27 Woodlawn Ave., Crafton, Pa.

GRIFFIN, Martin L. (Oct. 1, '04) Manager of Chemical & Electrochemical Depts., The Oxford Paper Co., Rumford, Me.

GRIFFITH, John R. (Feb. 23, '12) Res. Engineer, Norton Co., Niagara Falls, N. Y.; mailing address, 122 First St., Niagara Falls, N. Y.

GROENWALL, Assar (July 29, '10) Eng., Vasagatan 17, Stockholm, Sweden.

GRONNINGSATAR, Anton (Oct. 21, '16) Gen. Mgr., Kristiansand Nikkelraffineringsverk, Kristiansand S, Norway.

GROSSMANN, M. A. (Nov. 27, '14) Bureau of Standards, Div. VIII, Washington, D. C.

GROSVENOR, Dr. Wm. M. (June 1, '07) Chemists Bldg., 50 East 41st St., New York City.

GROTZINGER, John (July 27, '17) Box 123, Bay Point, Cal.

GROWER, Geo. G. (Nov. 5, '04) Coe Brass Mfg. Co.; res., 15 Colony St., Ansonia, Conn.

GRÜBNAU, G. M. (Oct. 21, '16) Los Cerrillos, Santa Fe County, New Mexico.

GRYMES, Elmer S. (Oct. 23, '14) Asst. Chief Chemist, U. S. Metals Ref. Co., Chrome, N. J.; res., 116 E. 7th Ave., Roselle, N. J.

GUDEMAN, Edward (Feb. 27, '09) Consulting Chemist and Scientific Expert, 903-4 Postal Telegraph Bldg., Chicago, Ill.

GUERBER, Arnold, J. (July 31, '08) Technologist, The Tungsten Products Co., Boulder, Colo.; mailing address, 2133 6th St.

GUESS, George A. (Aug. 5, '05) Professor of Metallurgy, Toronto University, Toronto, Canada.

GUESS, H. A. (Mar. 27, '14) Managing Director, Mining Dept., American Smelting and Refining Co., 120 Broadway, New York City.

GUINTHER, John (May 25, '17) Chem., Union Carbon Co. of Canada Ltd.; mailing address, Box 389, Welland, Ontario, Canada.

GUITERMAN, Kenneth S. (June 28, '12) care of Hendrickson & Co., 61 Broadway, New York City.

GUYE, Prof. Phillippe A. (Dec. 4, '02) 3 Chemin Bizot, Florrisant, Geneva, Switzerland.

HAANEL, Dr. Eugene (July 31, '07) Director of Mines, Dept. of Mines, Ottawa, Canada.

HABER, Prof. Dr. F. (Nov. 6, '02) Kaiser Wilhelm-Institut für Physikalische Chemie und Elektrochemie, Berlin-Dalem, Faradayweg 4, Germany.

HADFIELD, R. A. (July 6, '06) Manag. Dir., Hadfield Steel Fdy. Co., Ltd., Parkhead House, Sheffield, England.

HALCOMB, Chas. H. (May 5, '10) Halcomb & Davidson, Inc., Singer Bldg., 149 Broadway, New York City.

HALL, Arthur E. (April 29, '11) Gen. Supt., Am. Smelt. & Ref. Co., Omaha, Neb.

HALL, C. A. (July 25, '15) Pelham Court, Germantown, Philadelphia, Pa.

HALL, E. L. (Oct. 21, '16) Chief Eng., Portland Gas and Coke Co., 294 Yamhill St., Portland, Oregon.

HALL, Henry M. (Feb. 25, '11) Supt., U. S. Aluminum Co., Massena, N. Y.; P. O. Box 97, Massena, N. Y.

HALL, John H. (July 27, '17) Met. Eng., Taylor-Wharton Iron & Steel Co., High Bridge, N. J.

HALL, M. L. (May 24, '13) Apt. 20, 1615 Swann St., N. W., Washington, D. C.

HAMANN, A. M. (June 1, '15) E. E., Niagara Electrochemical Co., Niagara Falls, N. Y.; mailing address, 212 Sixth Street.

HAMBLY, Frederick J. (May 27, '11) Works Mgr., Electric Reduction Co., Ltd., Buckingham, Quebec, Canada.

HAMBUECHEN, Carl (Apr. 3, '02) Sec'y and Asst. Gen Mgr., American Carbon and Battery Co., East St. Louis, Ill.; mailing address, 4 Pennsylvania Ave., Belleville, Ill.

HAMILTON, E. H. (Oct. 22, '15) Metallurgist, The Consolidated Min. & Smelt. Co. of Canada, Ltd., Trail, B. C., Canada.

HAMISTER, Victor C. H. (Apr. 27, '12) 521 N. Henry St., Madison, Wis.

HAMOR, Wm. A. (Aug. 25, '17) Asst. Director, Mellon Inst.; mailing address, 1030 Victoria Ave., New Kensington, Pa.

HANCOCK, H. Lipson (Mar. 26, '10) General Manager, The Wallaroo & Moonta Mining & Smelting Co., Ltd., Moonta Mines, South Australia.

HANKS, A. A. (July 27, '17) Chem. and Met.; mailing address, 630 Sacramento St., San Francisco, Cal.

HANLEY, Herbert R. (Apr. 26, '17) Supt. Electrolytic Zinc Plant, Mammoth Copper Co., Kennett, Cal.

HANSCOM, Wm. W. (May 27, '11) Consulting Elec. Engr., 848 Clayton St., San Francisco, Cal.

HANSEN, C. A. (May 6, '05) 183 J St., Salt Lake City, Utah.

HANSON, Hugo H. (May 22, '14) Director, Eastern Mfg. Co., South Brewer, Me.
HARDCASTLE, Yellott F. (Oct. 22, '15) Supt. Pennsylvania Salt Mfg. Co., Wyandotte, Mich.
HARDIE, Chas. G. (Nov. 26, '15) Construction Dept., General Electric Co., 1001 Electrical Bldg., Buffalo, N. Y.
HARIG, Fred. C. (July 27, '17) Electrochemist, care of C. M. Hall Lamp Co., Rivard and Handcock St., Detroit, Mich.
HARPER, H. A. (Dec. 31, '15) Foreman, Tailing Retreatment Plant, care of Braden Copper Co., Rancagua, Chile.
HARPER, Dr. H. W. (Apr. 3, '02) Univ. of Texas, Austin, Texas; mailing address, 2216 Rio Grande St.
HARPER, John L. (Apr. 6, '07) Chief Eng., Hydraulic Power Co.; res., 148 Buffalo Ave., Niagara Falls, N. Y.
HARRIS, Jonathan W. (Sept. 26, '08) Research Chemist, Western El. Co., 463 West St., New York City.
HARRIS, Joseph W. (Apr. 3, '02) Patent Lawyer, 3105 18th St., N. W., Washington, D. C.
HART, Edw., Ph.D. (Aug. 7, '02) Prof. of Chemical Eng., Lafayette College, Easton, Pa.
HART, L. O. (Nov. 27, '09) Electrical Engineer, Driver Harris Wire Co., Harrison, N. J.; res., 232 Washington St., Hoboken, N. J.
HARTLEY, Robt. H. (Dec. 27, '07) Chemist, Hartley Bldg., Fourth Ave. and Smithfield Sts., Pittsburgh, Pa.
HARTWICK, Frank A. (Oct. 21, '16) 3476 Boulevard, Jersey City, N. J.
HARVEY, Frederic A. (Apr. 22, '15) Physicist, Solvay Process Co., care of Laboratory, Syracuse, N. Y
HASLWANTER, Chas. (Apr. 3, '02) 447 Spruce St., Richmond Hill, L. I., N. Y.
HASSLACHER, Jacob (Nov. 26, '07) Pres., Roessler & Hasslacher Chem. Co., 100 William St.; P. O. Box 1999, New York City.
HATCH, Israel (Oct. 28, '09) Asst. Superintendent, Elgin National Watch Co., Elgin, Ill.
HATZEL, J. C. (Apr. 3, '02) Pres. Hatzel & Buehler Inc., 373 Fourth Ave., New York City.
HAWKINS, Laurence A. (Apr. 27, '12) Electrical Engineer, Research Laboratories, General Electric Co., Schenectady, N. Y.
HAYES, Geo. W. (Mar. 26, '10) care of Marconi Wireless Tel. Co. of America, Aldene, N. J.
HAYES, Junius J. (Sept. 27, '16) Pleasant Grove, Utah Co., Utah.
HAYNES, Justin H. (Mar. 24, '16) Metallurgical Eng., Vindicator Con. Co., Independence, Colo.
HAYWARD, Robert F. (Jan. 29, '10) General Manager, Western Canada Power Co., Ltd., Vancouver, B. C., Canada.
HAZELETT, C. W. (May 2, '17) Storage Battery Eng., National; mailing address, 12816 Franklin Ave., Lakewood, Ohio.
HEATH, H. E. (Apr. 3, '02) 83 Court St., Newark, N. J.
HEATH, R. F. (Oct. 21, '16) Chem. Eng., care of Franklin Chem. Co., P. O. Box 740, Billings, Mont.
HEDDEN, Stanley E. (Feb. 24, '17) Foreman, Electroplater and Chem. Enameled Metals Co.; mailing address, 216 Fifth St., Aspinwall, Pa.
HEDIN, Joseph E. (Apr. 22, '15) General Supt., North American Pulp and Paper Co., Chandler, P. Q., Canada.
HEDLUND, Marten (Apr. 1, '05) Managing Dir., Gullspangs Elektrokemiska Aktienbolag, Gullspang, Sweden.
HEDSTROM, E. S. (Jan. 26, '17) E. E. and Mech. Eng., 38 Wood St., Toronto, Ont., Canada.
HEITMANN, Edward (Sept. 26, '08) Elec. Eng., care of Canadian Crocker-Wheeler Co., Ltd., Belleview Terrace, St. Catherines, Ont., Canada.
HELFENSTEIN, Dr. Alois (June 21, '11) Consulting Engineer, Bastiengasse 50, Vienna, XVIII, Austria.
HELFRECHT, A. J. (Mar. 24, '16) C. F. Burgess Labs., Madison, Wis.
HEMINGWAY, Frank (Nov. 26, '07) Pres. Hemingway & Co., Inc., No. 6, East Union Ave., Bound Brook, N. J.
HENDERSON, C. T. (Mar. 24, '16) Submarine Boat Corp., P. O. Box 456, Newark, N. J.
HENDERSON, Ernest G. (June 1, '15) Pres., care of The Canadian Salt Co., Windsor, Ont., Canada.
HENDERSON, John B. (Dec. 26, '13) Government Analyst, Brisbane, Queensland, Australia.
HENDRIE, G. A. (Nov. 27, '14) P. O. Box 36, Elizabeth, N. J.
HENDRY, W. Ferris (Nov. 26, '07) Engineer, 463 West St., New York City.
HENSEN, Emil (May 26, '10) 1200 State Ave., Coraopolis, Pa.
HERAEUS, Dr. phil. Wilhelm, Firm of W. C. Heraeus, Ltd., Hanau, Germany.

HERING, Carl (Apr. 3, '02) D.Sc., Consult. Elec. Eng., 210 S. Thirteenth St., Philadelphia, Pa.
HERRESHOFF, James B., Jr. (May 27, '14) Consult. Eng., 524 Beech St., Richmond Hill, Long Island, N. Y.
HERTY, C. H. (Oct. 22, '15) Editor, The Journal of Industrial & Engineering Chem., 35 East 41st St., New York City.
HERZ, Alfred (Feb. 27, '14) Testing Engineer, Public Service Co. of N. Illinois, Room 1322, 72 W. Adams St., Chicago, Ill.
HERZOG, G. K. (Feb. 27, '09) Electrometallurgical Co., Niagara Falls, N. Y.; mailing address, 8 Ericson Place.
HESS, Frederick M. (Nov. 27, '09) President and General Manager, Inyo Telephone Co., Bishop, Cal.
HESS, Henry (Feb. 25, '11) 308 Bailey Bldg., Philadelphia, Pa.
HESSOM, B. F., Jr. (May 26, '10) General Inspector, Duquesne Light Co., 435 Sixth Ave., Pittsburgh, Pa.; res., 136 Fifth St., Aspinwall, Pa.
HIBBARD, Henry D. (May 30, '08) Consult. Eng., 144 E. Seventh St., Plainfield, N. J.
HIBBERT, Harold (Apr. 22, '15) Research Chemist, care of Ralph L. Fuller Co., 2 Rector St., New York City.
HICKS, Edwin F. (May 26, '10) Chief Chemist, Victor Talking Machine Co., Camden, N. J.; res., 4837 Fairmount Ave., Philadelphia, Pa.
HIGASHI, Sentaro (Sept. 15, '15) Met. and El. Met. Eng., Ikuno Mine, Tajima, Japan.
HIGGINS, Aldus C. (Sept. 4, '02) Sec'y and Gen. Counsel, Norton Co., Worcester, Mass.
HILDEBRAND, Joel H. (Oct. 1, '12) 306 N. 35th St., Philadelphia, Pa.
HILL, Nicholas S., Jr. (Nov. 27, '09) Consult. Eng., 100 William St., New York City; res., 381 William St., East Orange, N. J.
HILL, Stafford (Jan. 27, '12) Electrician, No. 1, Bridge St., Stafford, England.
HILLEBRAND, W. A. (July 27, '17) E. E., Pacific Gas and Elec. Co.; mailing address, 445 Sutter St., San Francisco, Cal.
HILLIARD, John D. (Sept. 26, '08) Glen Falls, N. Y.
HILLS, Leander H. (June 30, '17) Student, Mass. Inst. of Tech.; mailing address, 30 Elm St., Gloucester, Mass.
HINCKLEY, A. T. (May 25, '12) Chemist, National Carbon Co.; res., 2 C Street, Niagara Falls, N. Y.
HINTON, Geo. B. (Nov. 24, '16) Consult. Met., 3 a Tabasca No. 69, Mexico, D. F., Mexico.
HIORTH, Albert C. F. (Dec. 31, '10) Josefinegade 13, Christiania, Norway.
HIORTH, Frederick V. L. (Dec. 26, '07) Electrochem. Eng., Josefinegade 19, 1, Christiania, Norway.
HIRSCH, Alcan (June 29, '07) Consulting Chemical Engineer, 50 E. 41st St., New York City.
HIRSCHLAND, Franz H., Dr. Ing. (Dec. 27, '07) Mgr., Goldschmidt Chem. Co., 60 Wall St., New York City.
HIRSHFELD, C. F. (May 25, '17) Chief of Res. Dept., Detroit Edison Co.; mailing address, 18 Washington Ave., Detroit, Mich.
HITCHCOCK, Fanny R. M. (May 1, '06) 4038 Walnut St., Philadelphia, Pa.
HITCHCOCK, Halbert K. (Oct. 2, '02) 5710 Bartlett St., Pittsburgh, Pa.
HITE, B. H. (Apr. 4, '03) W. Va. Exp. Station, Morgantown, W. Va.
HITNER, Harry F. (July 27, '17) Pittsburgh Plate Glass Co., 515 4th St., Oakmont, Pa.
HOBSBAWN, I. B. (July 27, '17) Consult. Tech. Chem., Messrs. Gibbs & Co., Valparaiso, Chile.
HOEFT, Eliot (July 27, '17) Efficiency Eng., American Chem. Products Co., 23 Liberty St., New York City.
HOFFMANN, John (Mar. 26, '15) Supt. of Copper Refinery, Balbach Smelt. & Ref. Co., 193 Smith St., Newark, N. J.
HOFFMANN, Rudolph (Dec. 26, '08) Prof. of Metallurgy, Kgl. Bergakademie, Clausthal, Germany; mailing address, 221 Bergstrasse.
HOGABOOM, Geo. B. (Feb. 25, '11) care of Scovill Mfg. Co., Waterbury, Conn.; mailing address, 561 Stanley St., New Britain, Conn.
HOGE, J. F. D. (Apr. 6, '11) The Maintenance Co., 417 Canal St., New York City; res., 509 W. 121st St., New York City.
HOLE, Ivar (Nov. 26, '15) Asst. Supt., Evje Smelter, Evje per Kristianssand S, Norway.
HOLLAND, Walter E. (Aug. 5, '05) Res. Eng., Philadelphia Storage Battery Co., Ontario and C St., Philadelphia, Pa.
HOLLER, H. D. (Nov. 26, '15) Asst. Chemist, Bureau of Standards, Washington, D. C.
HOLMES, Major E. (Oct. 23, '14) Chemcal Eng., Nat. Carbon Co.; mailing address, 1459 Arthur Ave., Cleveland, Ohio.

HOLSTEIN, Leon Stuart (Sept. 28, '12) N. J. Zinc Co., Palmerton, Pa.; mailing address, 477 Columbia Ave., Palmerton, Pa.
HOLTON, Fred. A. (Apr. 2, '04) Chemist in Patent Causes, 700 10th St., N. W., Washington, D. C.
HOMMEL, Rudolf P. (June 2, '16) Asst. Secretary. American Electrochemical Society, Lehigh University, Bethlehem, Pa.
HOOD, B. B. (July 23, '15) Asst. to the Supt., U. S. Metals Refining Co., Chrome, New Jersey.
HOOKER, Albert H. (Feb. 27, '09) Hooker Electrochemical Co., Niagara Falls, N. Y.
HOPKINS, Geo. A. (Feb. 25, '11) Metallurgist, Carnegie Steel Co., Munhall, Pa.; res., 464 Swissvale Ave., Wilkinsburg, Pa.
HORNSEY, John W. (May 5, '10) Consulting Engineer, Summit, N. J.
HORSCH, Wm. G. (Apr. 26, '13) Graduate Student, Dept. of Chem., Univ. of California, Berkeley, Cal.
HOSFORD, Wm. F. (Oct. 22, '15) Eng. of Methods, Western Electric Co., Hawthorne Works, Chicago, Ill.
HOSKINS, Wm. (Apr. 3, '02) Suite 2009, Harris Trust Bldg., 111 W. Monroe St., Chicago, Ill.
HOUGH, Arthur (May 1, '06) Curtis & Harvey, Ltd., 400 St. James St., Montreal, Canada.
HOWARD, Geo. M. (Apr. 3, '02) 19th St. and Allegheny Ave., Philadelphia, Pa.
HOWARD, Henry (Apr. 3, '02) 36 Amory St., Brookline, Mass.
HOWARD, L. E. (May 29, '09) Metallurgist, Simonds Mfg. Co., Lockport, N. Y.; mailing address, 493 East Ave.
HOWARD, Prof. S. Francis (Apr. 3, '02) Prof. of Chem., Norwich Univ., Northfield, Vermont.
HOWARD, W. D. M. (Sept. 27, '16) Consult. Eng., Industrial Development Syndicate; mailing address, 1074 Union St., San Francisco, Cal.
HOWE, David (Sept. 27, '16) Foreman, Reed & Barton, 16 Rockland St., Taunton, Mass.
HOWE, Prof. Henry M. (Aug. 7, '02) Professor of Metallurgy, Columbia University, New York City; res., Broad Brook Road, Bedford Hills, N. Y.
HUBLEY, Warren F. (May 24, '13) Pres., American Transformer Co., 145 Miller St., Newark, N. J.
HULETT, Geo. A. (Apr. 2, '04) Princeton Univ., Princeton, N. J.
HUMBERT, Ernest P. (Mar. 27, '09) Box 26, Welland, Ontario, Canada.
HUNT, A. M. (Apr. 3, '02) Consult. Eng., 55 Liberty St., New York City.
HUNTER, J. Vincent (Jan. 28, '11) Gen. Foreman of Shops, Twin Cities St. Railways Co.; mailing address, 152 Arthur Ave., Minneapolis, Minn.
HUNTER, M. A. (Apr. 29, '11) Assistant Professor, Physics and Electrochemistry, Rensselaer Polytechnic Institute, Troy, N. Y.; res., 8 Whitman Court.
HUNTOON, Louis D. (May 27, '14) 115 Broadway, New York City.
HURUM, Frederick (Nov. 26, '15) Mass. Inst. of Tech., Westmoreley Court, Cambridge, Mass.
HUTCHINGS, Chas. F. (June 27, '13) Gen. Mgr. North American Chemical Co., Bay City, Mich.
HUTCHINS, Otis (Jan. 28, '11) Mentz Apartments, Niagara Falls, N. Y.
HYDE, Edward P. (Oct. 29, '08) Director, Nela Res. Lab., National Lamp Wks. of General Electric Co., Nela Park, Cleveland, Ohio.
HYDE, Reed W. (Oct. 22, '15) Res. Chem., American Smelt. and Refining Co., Murray, Utah.
IGARASHI, Tadao (Apr. 22, '15) Eng., Electro Iron Reduction Co., Tokio, Japan; mailing address, 30 Takanawa Minami-cho, Shiba-ku, Tokyo, Japan.
IMLAY, Lorin E. (Dec. 31, '09) Superintendent, The Niagara Falls Power Co., Niagara Falls, N. Y.
INGALLS, Walter Renton (June 29, '07) 10th Ave. at 36th St., New York City.
INUI, Kyujiro (Oct. 26, '17) Mitsubishi Co., 120 Broadway, New York City.
IONIDES, S. A. (Apr. 26, '17) Electrochem. Eng., 802 Equitable Bldg., Denver, Colo.
ISHIKAWA, Ichiro (Oct. 22, '15) Chem. Eng., 357 Nishigahara, Takinogawa, Tokyo, Japan.
IWAI, Kyosuke (May 26, '10) Gen. Supt., Yusenji Copper Mine, Komatsu-Machi, Nomigun, Ishikawaken, Japan.
JACK, Geo. B., Jr. (Oct. 21, '16) care of Metallurgical Dept., Chalmers Motor Co., Detroit, Mich.
JACKSON, Alf. Geo. (July 30, '09) Manager, Synchronome Electrical Co. of Australasia, Ltd.; res., 65 Ann St., Brisbane, Queensland, Australia.
JACOB, Arthur (Dec. 30, '16) Sterndale, Hatch End, Middlesex, England.
JACOBSEN, Frederik (Sept. 24, '10) Consulting Engineer, Kleven 22, Stavanger, Norway.
JACOBSON, B. H. (Mar. 24, '16) Research El. Met., Hooker Eelectrochemical Co., Niagara Falls, N. Y.; mailing address, 229 Third St.
JACOBY, Henry E. (Oct. 21, '16) Mech. Eng., 436 Convent Ave., New York City.

JAMES, Dr. J. H. (Apr. 3, '02) Prof. of Chem. Eng., Chem. Dept., Carnegie Technical School, Pittsburgh, Pa.

JAMES, Wm. F. (Oct. 21, '16) Sales Eng., Westinghouse Elec. and Mfg. Co., 227 W. Mt. Pleasant Ave., Philadelphia, Pa.

JARVIS, Ernest G. (Sept. 27, '16) Chemist and Met., care of McNab & Harlin Mfg. Co., Paterson, N. J.

JENISTA, Prof. Geo. J. (Sept. 24, '10) Graduate Dept., Univ. of Chicago, Chicago, Ill.; mailing address, 3160 Abbott Court.

JENNISON, Herbert C. (Feb. 29, '08) with the American Brass Co., Ansonia, Conn.; mailing address, P. O. Box 600.

JEPPSON, Geo. N. (Sept. 4, '02) Norton Co., Worcester, Mass.

JESPERSON, C. M. (Aug. 25, '16) Sec'y-Treas., care of Southern Manganese Corp., Anniston, Ala.

JINGUJI, Genjiro (May 25, '17) Consult. Eng., 682 Academy St., New York City.

JOHANSEN, G. H. (Aug. 27, '09) Civil Eng., Gen. Sec'y of Norwegian Water Power Co., Bankpladsen 1, Kristiania, Norway.

JOHNS, Morgan J. (June 25, '09) care of Mount Morgan Gold Mining Co., Mount Morgan, Queensland, Australia.

JOHNSON, Arden R. (June 2, '06) Director of Arden Johnson Labs., Rooms 718-720, 9 S. Clinton St., Chicago, Ill.

JOHNSON, Joseph A. (May 25, '12) Electrical Engineer, Ontario Power Co., Box 333, Niagara Falls, N. Y.

JOHNSON, J. E., Jr. (June 1, '15) Consult. Eng. & Met., 52 William St., New York City.

JOHNSON, Woolsey McA. (Apr. 3, '02) Pres., The Continuous Zinc Furnace Co., 599 Broad St., Hartford, Conn.

JOHNSTON, Frederick A. (Apr. 24, '09) Supt., Assay and Metallurgical Division, The S. S. White Dental Mfg. Co., Prince Bay, S. I., N. Y.

JOHNSTON. Wm. A. (Oct. 10, '03) Supt. of Mfg., S. S. White Dental Mfg. Co., Prince Bay, S. I., N. Y.

JONES, Abner C. (May 25, '17) Chem. & Metallurgist, care of Otis Elevator Co., Buffalo, N. Y.

JONES, George H. (Oct. 29, '10) Power Engineer, Commonwealth Edison Co., Chicago, Ill.; res., 279 Keystone Ave., River Forest, Ill. (Oak Park, P. O.)

JONES, Grinnell (Nov. 26, '10) Chem. Expert, U. S. Tariff Com., 1322 New York Ave.; mailing address, 712 Allison St., N. W., Washington, D. C.

JONES, G. W. (Nov. 23, '17) Chemist, U. S. Bureau of Mines, 5762 Baum Blvd., Pittsburgh, Pa.

JONES, Harold (May 29, '09) Metallurgist, The St. John del Rey Gold Mining Co., Villa Nova de Lima, Minas Geraes, Brazil.

JORGENSEN, E. L. (Aug. 25, '16) Met. Eng., Guggenheim Bros.; mailing address, care of Chile Exploration Co., Chuquicamata, Chile.

KAHLENBERG, Louis, Ph.D. (Apr. 3, '02) Prof. of Phys. Chem., Univ. of Wis., Madison, Wis.; res., 234 Lathrop St.

KAKU, Juroku (May 25, '17) Nikko Copper Refining Plant, Nikko, Japan.

KALMUS, Dr. Herbert T. (May 25, '12) Vice-Pres. and Treas., The Exolon Co., Cambridge, Mass.; mailing address, Kalmus, Comstock & Wescott, Inc., Engineers and Industrial Res. Chemists, 156 Sixth St., Cambridge, Mass.

KAMEYAMA, Naoto (Dec. 31, '15) 10 Nishikatamachi, Hongo, Tokyo, Japan.

KÄMMERER, Jacob A. (Oct. 28, '09) President and Gen. Mgr., Wood Products Co. of Canada, Toronto, Canada; res., 87 Jameson Ave.

KAMMERHOFF, H. H. M. (Sept. 27, '16) Mgr., Coal Tar Products Div. of Thos. A. Edison Inc., Bloomfield, N. J.

KANEKO, Kiosuke (Sept. 27, '13) Prof. of Met., College of Engineering, Imperial Univ. of Kyushu, Fuknoka, Japan.

KAO, Takang (Dec. 30, '16) Mech. Eng., care of American Whaley Engine Co., 136 Federal Bldg., Boston, Mass.

KATSURA, Prof. Benzo (May 29, '09) Professor of Metallurgy, The Imperial Tokyo University, Tokyo, Japan; res., 58 Sendaki-Machi, Hongo-Ku, Tokyo.

KAUFFMANN, F. A. (Aug. 25, '16) Chemist, The Roessler & Hasslacher Chem. Co., 160 High St., Perth Amboy, N. J.

KAWAMURA, Takeshi (June 28, '12) Kenjiko Iron Works, Mitsubishi Goshi Kaihsa, Kenjiho, Koshu-gun, Kokai-do, Korea, Japan.

KAWIN, Chas. C. (Nov. 6, '10) Pres., Chas. C. Kawin Company, Chicago, Ill.; res., 431 S. Dearborn St.

KEEN, Wm. Herbert (Aug. 25, '16) Gen. Mgr., Chemical Products Co., Washington, D. C.; mailing address, 3210 34th St., N. W.

KEENEY, Robt. M. (Nov. 24, '11) Mgr., The Ferro Alloy Co., Denver, Colo.; mailing address, 1418 Elizabeth St.

KEFFER, Frederick (Feb. 27, '09) Cons. Eng. and Geologist, 610 Hutton Bldg., Spokane, Wash.

KEITH, Dr. N. S. (April 3, '02) 350 Bullitt Bldg., Philadelphia, Pa.

KELLEHER, James (Feb. 24, '17) Asst. Eng., with R. Turnbull; mailing address, care of R. Turnbull, Esq., Welland, Ont., Canada.

KELLER, Ch. A. (June 25, '09) Gen'l Mgr., Keller-Leleux Cie., 3 Rue Vignon, Paris, France.
KELLER, Ed. (Apr. 3, '02) 365 West 56th St., New York City.
KELLER, Oran (Oct. 23, '14) Chemist, U. S. Metals Ref. Co., 253 George St., New Brunswick, N. J.
KELLY, Dr. John F. (Apr. 3, '02) Stanley Elec. Mfg. Co.; res., 284 W. Housatonic St., Pittsfield, Mass.
KELLOGG, Harry W. (Jan. 29, '09) Gen'l Mgr., National Electrolytic Co., Niagara Falls, N. Y.
KEMERY, Philo (May 29, '09) Metallurgical Engineer, Crescent Works, Crucible Steel Co. of America, Pittsburgh, Pa.
KEMMER, Frank R. (Feb. 25, '11) The Aluminum Co. of America, Whitney, North Carolina.
KEMPER, David A. (Oct. 21, '16) Chemist, Marden, Orth & Hastings, Newark, N. J.; mailing address, 1144 President St., Brooklyn, N. Y.
KENAN, Wm. R. (Apr. 3, '02) 433 Locust St., Lockport, N. Y.
KENNEDY, A. M. (Sept. 27, '16) Staff Eng., Edison Lab., Orange, N. J.
KENNEDY, J. J. (May 9, '03) Enigneer, 52 Broadway, New York City.
KENNEDY, T. O. (Apr. 26, '16) Gen. Mgr., The Mass. Electric and Gas Co., Massillon, Ohio.
KENRICK, Frank B. (Apr. 26, '13) Associate Prof. Chem., Univ. of Toronto, Toronto, Canada; res., 77 Lonsdale Road.
KENT, Jas. M. (Sept. 4, '03) Consult. Eng. and Teacher and Engineer of Applied Steam and Electricity, Manual Training School, 2446 Harrison St., Kansas City, Mo.
KERN, Ed. F., Dr. (Apr. 4, '03) care of Dept. of Metallurgy, Columbia Univ., New York City.
KERN, P. E. (Nov. 24, '11) 419 N. Central Park Blvd., Chicago, Ill.
KEYES, Donald B. (Oct. 3, '17) Asst. in Chemistry, Univ. of California; mailing address, 2606 Bancroft Way, Berkeley, Cal.
KIER, Samuel M. (Oct. 29, '08) Pres., Kier Firebrick Co., 2243 Oliver Bldg., Pittsburgh, Pa.
KINGSLEY, Edw. D. (Oct. 26, '17) Pres. Electro Bleaching Gas Co. and Niagara Alkali Co.; mailing address, 18 E. 41st St., New York City.
KINNEAR, H. B. (Aug. 25, '16) Metallurgist, Whitaker-Glessner Co.; mailing address, 1918 Summit St., Portsmouth, Ohio.
KINTER, Geo. R. (Mar. 24, '16) 1403 State St., Harrisburg, Pa.
KISHI, Keijiro (Nov. 21, '08) Chief Eng., El Dept., Shibaura Eng. Wks., No. 1, Shinhamacho, Kanasugi, Shibaku, Tokyo, Japan.
KISSOCK, Alan (Sept. 20, '11) 63 Wall St., New York City; mailing address, P. O. Box R, Tucson, Arizona.
KITSEE, Dr. Isador (Apr. 3, '02) 306 Stock Exchange Place, Philadelphia, Pa.
KLEINFELDT, Henry F. (Apr. 22, '15) Sec'y, Abbe Engineering Co., 121 Park Ave., Newark, N. J.
KLIPSTEIN, Ernest C. (Apr. 3, '02) 122 Pearl St., P. O. Box 2833, New York City; res., 116 Prospect St., East Orange, N. J.
KLOWMAN, Henning (Oct. 3, '17) Mgr. Elec. Steel Plant, Messrs. Hadfields, Ltd., 24 Wilson Road, Hunters Bar, Sheffield, England.
KLUGH, B. G. (Apr. 7, '06) P. O. Box 21, Anniston, Ala.
KNAPP, Geo. O. (Nov. 6, '02) 42d St. Bldg., New York City.
KNIFFIN, L. M. (Aug. 25, '16) Asst. Supt., U. S. Smelting, Ref. and Min. Co., 55 Congress St., Boston, Mass.; mailing address, P. O. Box 385.
KNIGHT, Frank P. H. (Feb. 25, '11) Electrician, Chemist, Inventor, Knight Ecker Co. Labs., Keokuk, Iowa; res., 1015 Blondeau St.
KNOEDLER, E. L. (Dec. 30, '16) Gen. Supt., Welsbach Co.; mailing address, 110 Brown St., Gloucester City, N. J.
KNOX, Lester B. (Mar. 24, '16) Latrobe Elec. Steel Co., Latrobe, Pa.
KOEHLER, Wm. (Nov. 5, '04) E. 792 Lakeview Road, N. E., Cleveland, Ohio.
KOERING, Bruno K. (Sept. 27, '16) Pres. and Gen. Mgr., Koering Cyanide Process Co.; mailing address, 220-224 Dooley Block, Salt Lake City, Utah.
KOERNER, Walter E. (May 27, '14) Electrochemist and Metallurgist, General Elec. Co.; mailing address, 255 Amherst St., East Orange, N. J.
KOETHEN, Frederick L. (Jan. 29, '10) Sales Eng. and Supt., Lubricant Works, International Acheson Graphite Co., Niagara Falls, N. Y.; mailing address, 1960 Whitney Ave.
KOHLER, Hy. L. (Aug. 31, '07) Metallurgist, Scullin Steel Co., St. Louis, Mo.; mailing address, 3322 Holliday Ave.
KOHN, Milton M. (May 29, '09) Mgr., Multiple Unit Electric Co., 136 Liberty St., New York City.
KOKATNUR, V. R. (Oct. 26, '17) Organic Chem., Castner Electrolytic Alkali Co., Niagara Falls, N. Y.
KOLKIN, T. L. (Oct. 27, '11) Mgr., A/s Vadheim Elektromiske Fabriker, Vadheim, Sogne, Norway.

KOONTZ, John A., Jr. (Sept. 27, '16) E. E., Great Western Power Co., 221 Bryant St., Palo Alto, Cal.
KOPPITZ, C. G. (Oct. 26, '17) Consult. Elec. Eng., Ry. & Industrial Eng. Co., Greensburg, Pa.
KOWALKE, O. L. (Aug. 3, '06) Assoc. Prof. of Chem. Eng., Chem. Eng. Bldg., Univ. of Wis., Madison, Wis.
KRAMER, Lewis B. (Mar. 24, '16) 109 Carsonia Ave., Mt. Penn, Pa.
KRANZ, Wm. G. (Apr. 29, '11) Vice-Pres., charge of Manufacturing, National Malleable Castings Co., 7706 Platt Ave., Cleveland, Ohio.
KRAUS, Ernest (Sept. 27, '13) 433 Gregory Ave., Weehawken, N. J.
KRAUSE, Walter B. (Apr. 24, '14) care of American Steel Foundries, Chester, Pa.
KREJCI, Milo W. (May 27, '09) Asst. Supt., Great Falls Reduction Works, Anaconda Copper Mining Co., Great Falls, Mont.; mailing address, 15 Smelter Hill.
KREMERS, J. G. (July 31, '07) 906 Shepherd Ave., Milwaukee, Wis.
KROG, Karl M. (June 30, '16) Shift Supt., Electrolytic Tank House, care of Chile Exploration Co., Chuquicamata, Chile.
KROYER, John M. (Aug. 25, '17) Pres. and Gen. Mgr., Samson Sieve-Grip Tractor Co., Stockton, Cal.; mailing address, 300 E. Acacia St., Stockton, Cal.
KUNZ, Geo. F., Ph.D., D. Eng. (Sept. 28, '07) Vice-Pres. Tiffany & Co., 405 Fifth Ave., New York City.
KUTZ, Milton (June 1, '15) Mgr., The Roessler and Hasslacher Chem. Co., 941 Drexel Bldg., Philadelphia, Pa.
KWANG, Kwong Yung, D. Eng. (Apr. 24, '09) Engineer and Director, Lincheng Mines, Lincheng, Chihli Province, Kin-Han Railway, via Pekin, N. China.
KYLE, T. D. (Apr. 3, '02) 106 E. 5th St., Leadville, Colo.
LACROIX, Henry (Mar. 3, '06) Eng., Usine de Degrossissage d'or, Geneve, Switzerland.
LAFORE, J. A. (Apr. 3, '02) Merion Paper Co., Bellevue Court Bldg., Philadelphia, Pa.; res., Wister Road, Ardmore, Pa.
LAIB, Walter (May 22, '14) Supt. and Chief Chem., The Ohio Salt Co., Rittman, Ohio; mailing address, Rittman, Wayne Co., Ohio.
LAISE, Clemens A. (Dec. 30, '16) Chem. Eng., Independent Lamp and Wire Co.; mailing address, 37 Duer Place, Weehawken, N. J.
LAMB, Arthur B. (Dec. 27, '07) Chemical Lab., Harvard College, Cambridge, Mass.
LAMKER, H. G. (May 24, '13) Johnson Bronze Co., Newcastle, Pa.
LAMOUREUX, Ernest (Apr. 26, '17) Saks Representative, Munning-Loeb Co.; mailing address, 844 Aldine Ave., Chicago, Ill.
LANDAU, Alfred (Apr. 22, '15) Pres., Canadian Carbon Co., Ltd.; mailing address, United Battery Corp. of N. Y., Woolworth Bldg., New York City.
LANDIS, Walter S. (Dec. 4, '02) Chief Technologist, American Cyanamid Co., New York; mailing address, East 31st St., Beechurst, L. I., New York.
LANDOLT, Dr. Hans (Feb. 5, '03) Turgi, Switzerland.
LANDOLT, P. E. (Nov. 27, '14) 1st Lieut., Ordnance R. C., Division T, Ordnance Dept., Washington, D. C.
LANE, Henry M. (May 29, '09) Trussed Concrete Bldg., Detroit, Mich.
LANGFORD, Frank (May 29, '09) 1112 J St., Eureka, Humboldt County, Cal.
LANGMUIR, Irving (June 29, '07) Research Lab., Gen'l Elec. Co., Schenectady, N. Y.
LANGTON, John (Apr. 3, '02) Consult. Eng., 233 Broadway, New York City.
LANSING, C. N. (Nov. 26, '15) Eng. on Cells, Hooker Electrochemical Co., Niagara Falls, N. Y.
LARCHAR, Arthur B. (Apr. 3, '02) Penobscot Chem. Fibre Co., Great Works, Me.
LASS, W. P. (Mar. 27, '09) Speel River Electrochemical Co., Juneau, Alaska.
LATHROP, L. H. (Sept. 26, '08) General Supt., Ironwood & Bessemer Ry. & Light Co., Ironwood, Mich.
LAUGHLIN, H. Hughart (May 29, '09) Elec. Engr., Jones & Laughlin Steel Co., 2709 Carson St., Pittsburgh, Pa.; res., 5023 Bayard St.
LAVENE, H. A. (Nov. 26, '15) Chemist, International Acheson Graphite Co., Niagara Falls, N. Y.; mailing address, 170 Buffalo Ave.
LAVINO, Edward J. (Nov. 26, '07) E. J. Lavino & Co., Importers of Ferro-Alloys, Bullitt Bldg., Philadelphia, Pa.
LAWRENCE, J. N. (Feb. 23, '12) 344 West Ave., Buffalo, N. Y.
LAY, J. Tracy (Nov. 26, '10) Graduate Student, University of Pennsylvania, Philadelphia, Pa.; res., 4015 Pine St.
LEA, John (Oct. 21, '16) Chem. and Met., with H. B. Howland; mailing address, 5410 Normandie Ave., Los Angeles, Cal.
LEACH, Edwin R. (Feb. 25, '11) Mgr. Island Copper Co., 716 Security Bank Bldg., Oakland, Cal.; mailing address, 217 Hillside Ave., Piedmont, Cal.
LEAVITT, Wm. F. B. (Mar. 26, '10) care of C. W. Leavitt & Co., 30 Church St., New York City.
LEBHERZ, John (Mar. 24, '16) President, Frontier Brass Foundry, 937-44 Elmwood Ave., Niagara Falls, N. Y.
LE BOUTILLIER, Clement (July 31, '08) Chem. and Met., High Bridge, N. J.
LEDOUX, Albert R. (July 25, '13) Eng. and Chemist. Pres., Ledoux & Co., Inc., 99 John St., New York City.
LEE, Harry R. (Dec. 2, '05) Holcomb's Rock, Va.
LEE, I. E. (Mar. 24, '16) Instructor in Chemistry, University of Rochester, N. Y.

LEFFEL, C. E. (Sept. 25, '14) Supt., The Spirella Co., Inc., Box 127, Niagara, Falls, N. Y.
LEFFLER, Johan A. (Sept. 24, '10) Technical Gen. Director, Gimo-Österby Bruks Aktiebolag, Osterby Bruk, Dannemora, Sweden.
LEGRAND, Charles (Mar. 24, '16) Consult. Eng., Phelps Dodge Co., mailing address, P. O. Box EE, Douglas, Arizona.
LEMBERG, Max (Apr. 6, '11) Chief Engr., Vulcan Detinning Co., Sewaren, N. J.; res., Woodbridge, N. J.
LENSCHOW, Hans H. (May 25, '17) Chem., Kristianssand's Nikkelraffineringsverke, Norway.
LEONARD, Grover M. (Sept. 27, '16) Store Keeper, The Jeffrey Mfg. Co.; mailing address, 1225 Wesley Ave., Columbus, Ohio.
LESLIE, E. H. (Sept. 27, '16) U. S. Industrial Alcohol Co., 27 William St., New York City.
LEVY, Gaston J. (Aug. 25, '17) Chem. Eng., Leslie Salt Ref. Co.; mailing address, 619 Laurel Ave., San Mateo, Cal.
LEWIS, Jonathan D. (May 29, '09) Chemist, Edgar Thompson Works, Carnegie Steel Co.; mailing address, 710 Middle St., Braddock, Pa.
LICHTHARDT, G. (Apr. 3, '02) 18th and M Sts., Box 510, Sacramento, Cal.
LIDBURY, F. Austin (Aug. 7, '03) Works Mgr., Oldbury Electrochem. Co.; res., 33 Sugar St., Echota, Niagara Falls, N. Y.
LIEBMANN, Dr. A. J. (Aug. 26, '10) 203 W. 81st St., New York City.
LIEBSCHUTZ, Morton (Jan. 28, '11) Analytical Chemist, The Balbach Smelt. and Ref. Co., Newark, N. J.; res., 719 De Graw Ave.
LIHME, C. B. (Nov. 27, '09) Director Matthiessen & Hegeler Zinc Co., La Salle, Ill.; mailing address, 1200 Lake Shore Drive, Chicago, Ill.
LILJA, S. G. (Oct. 21, '16) Eng., Hamilton & Hansell; mailing address, 6 Malmtorgsgatan, Stockholm, Sweden.
LILJENROTH, F. G. (Dec. 31, '15) Consult. Eng., care of Engineering Dept., duPont Co., Wilmington, Del.
LINCOLN, Dr. A. T. (Nov. 6, '02) Prof. of Physical Chemistry, Rensselaer Polytechnic Institute, Troy, N. Y.; mailing address, 1625 Tibbitts Ave.
LINCOLN, Edwin S. (Sept. 26, '08) Consult. Eng., 534 Congress St., Portland, Maine.
LINCOLN, P. M. (Apr. 3, '02) 6830 Thomas St., Pittsburgh, Pa.
LIND, S. C. (July 30, '09) Chem. in Radioactivity, U. S. Bureau of Mines, Golden, Colo.
LINDBERG, Sven C. son (Sept. 27, '13) Tech. Mgr., Stierns Aktiebolag, Vddeholm, Sweden.
LINDEN, H. E. (May 2, '17) Hydro-Elec. Eng., Sec'y-Treas., Beckman and Linden Eng. Corp., 604 Balboa Bldg., San Francisco, Cal.
LINDSAY, Dr. Chas. F. (June 1, '07) British War Mission, Purchasing Dept., 120 Broadway, New York City.
LISCOMB, Fred. J. (Feb. 25, '16) Salesman, Hanson & Van Winkle Co., 6066 Nickerson Ave., Chicago, Ill.
LITTLE, Arthur D. (Apr. 1, '05) Chem. Eng. and Expert, 30 Charles River Road, Cambridge, Mass.
LITTLE, Wm. T. (Mar. 24, '16) Research Dept., Al. Co. of America, Box 35, Massena, N. Y.
LJUNGH, Hjalmar (Mar. 27, '09) Florogatan 21, Stockholm 5, Sweden.
LLOYD, M. G., Ph.D. (Apr. 3, '02) Elec. Eng., Bureau of Standards, Washington, D. C.
LLOYD, Sherman C. (Sept. 26, '08) 1402 Gilpin Ave., Wilmington, Del.
LLOYD, Stewart J. (Oct. 28, '09) Professor of Metallurgy and Chem., University of Alabama, University, Ala.
LOEBELL, Henry O. (Aug. 28, '14) Industrial Heating Expert, Henry L. Doherty & Co., 60 Wall St., New York City.
LOEFFLER, Geo. O. (Mar. 24, '16) Metallurgist, Carbon Steel Co.; mailing address, Bellefield Dwellings, Pittsburgh, Pa.
LOF, E. A. (Feb. 24, '17) Elec. Eng., Power and Mining Eng. Dept., General Electric Co., Schenectady, N. Y.
LOHR, J. M. (May 24, '13) Alloy Chemist, 1616 W. Grand Blvd., Detroit, Mich.
LOKEN, Rikard (Nov. 26, '15) Chemist, Kristianssand Nikkelraffineringsverk, Kristianssand S, Norway.
LONG, Geo. E. (Jan. 28, '08) 732 W. Washington St., Napoleon, Ohio.
LOVE, Edward G. (May 29, '09) Chief Chemist, Consolidated Gas Co., 130 E. 15th St., New York City.
LOVEJOY, D. R. (Apr. 3, '02) U. S. E. M. Co., 221 W. 33d St., New York City.
LOVELACE, B. F. (Oct. 27, '11) Associate Prof. of Chemistry, Johns Hopkins University, Baltimore, Md.
LOW, Frank S. (Apr. 26, '16) Chem. Eng., care of Niagara Smelting Corp., Niagara Falls, N. Y.
LOWE, Russell E. (Jan. 26, '17) Chem., The FitzGerald Labs., Inc., 170 Buffalo Ave., Niagara Falls, N. Y.
LUBOWSKY, Simon J. (Apr. 26, '17) Head, Chem. Dept., Goldschmidt Thermit Co., 92 Bishop St., Jersey City, N. J.
LUKENS, Hiram S. (Feb. 27, '14) Asst. Prof. of Chem., John Harrison Lab. of Chemistry, Univ. of Pa., Philadelphia, Pa.

LUNDGREN, Harald (Aug. 25, '11) 7742 Marquette Ave., Chicago, Ill.
LUNN, Ernest (Jan. 29, '09) The Pullman Co., Room 801, Pullman Bldg., Chicago, Ill.
LYMAN, James (Apr. 3, '02) Firm of Sargent & Lundy, 1412 Edison Bldg., Chicago, Ill.
LYON, Dorsey A. (Feb. 27, '09) Supervisor of Experiment Stations, Bureau of Mines, Washington, D. C.
LYONS, Henry N. (Oct. 21, '16) 25 Jaggar Ave., Flushing, N. Y.
LYSTER, T. L. B. (Nov. 26, '15) Eng., Hooker Electrochemical Co., Niagara Falls, N. Y.
LYTLE, Lloyd B. (June 30, '17) E. E., with Consolidated Min. and Smelt. Co. of Canada, Ltd.; mailing address, Box 18, Trail, B. C., Canada.
MAC DONALD, Jas. A. (Aug. 7, '03) Vice-Pres., United Verde Copper Co., 26 Exchange Place, New York City.
MAC DOUGALL, A. J. (Oct. 26, '17) Mech. Eng., Toronto Power Co.; mailing address, 12 Adelaide St., East, Toronto, Ontario, Canada.
MAC GREGOR, Frank S. (Sept. 28, '07) Development Dept., Room 452, Du Pont Bldg., Wilmington, Del.
MAC GREGOR, Walter (Jan. 29, '10) 125 12th St., Northwest, Canton, Ohio.
MAC INNES, Duncan A. (Jan. 25, '13) Chemical Research Lab., Mass. Inst. of Technology, Cambridge, Mass.
MACK, E. L. (Dec. 31, '15) Graduate Student, Cornell Univ., Morse Hall, Ithaca, N. Y.
MAC MAHON, Jas. (Aug. 7, '02) Bleaching Powder Supt., Castner Electrolytic Alkali Co., Niagara Falls, N. Y.
MAC MAHON, J. Douglas (Aug. 25, '16) Asst. in Res. Dept., Norton Co.; mailing address, 15 Ericsson Place, Niagara Falls, N. Y.
MAC MILLAN, J. R. (Oct. 23, '14) Chemist, Niagara Falls Alkali Co., 428 11th St., Niagara Falls, N. Y.
MAC NUTT, Barry, E.E., M.S. (Apr. 3, '02) Phys. Lab., Lehigh Univ., Bethlehem, Pa.
MAC RAE, Duncan (Oct. 26, '17) Chief Chem., Chem. Lab., Westinghouse Lamp Co., Bloomfield, N. J.
MADSEN, Chas. P. (Oct. 22, '15) Consult. Engineer, 44 Walnut St., Newark, N. J.
MAGNUS, Benjamin (Apr. 3, '02) Consult. Eng., care of Adolph Lewisohn & Sons, 61 Broadway, New York City.
MAHLMAN, Lieut. O. L. (Jan. 26, '17) care of Astoria Light, Heat and Power Co., Lawrence Point, Astoria, Long Island, N. Y.
MAIER, Chas. G. (Dec. 31, '14) Res. Chemist, care of Phelps, Dodge Co., 99 John St., New York City.
MAILLOUX, C. O., D.Sc. (Apr. 3, '02) Consult. Elec. Eng., 20 Nassau St., New York City.
MAIN, Wm. (Apr. 3, '02) Piermont, Rockland Co., N. Y.
MAINWARING, Wm. D. (Apr. 24, '14) Production Engineer, 866-70 Rockefeller Bldg., Cleveland, Ohio.
MALINOVSZKY, Andrew (Oct. 21, '16) Chem. Eng., Malinite Co., 22 N. Douglas Ave., Belleville, Ill.
MALM, John L. (Aug. 5, '05) Electrochemical Eng., 323 1st Nat. Bank Bldg., Denver, Colo.
MANAHAN, Paul R. (Apr. 30, '08) Rubber Insulated Metals Corp., Plainfield, N. J.
MANN, Chas. A., Ph.D. (Feb. 24, '17) Prof. of Chem. Eng.; mailing address, Chem. Eng. Dept, Iowa State College, Ames, Iowa.
MANN, Wallace W. (May 5, '10) Foreman, Battery Department, The National Carbon Co., Cleveland, Ohio; res., 1369 Summit Ave., Lakewood, Ohio.
MANTIUS, Otto (Jan. 28, '08) Consult. Eng., Woolworth Bldg., New York City.
MARIE, Charles, Dr. des Sciences (Jan. 8, '04) 9 rue de Bagneux, Paris, VI, France.
MARSH, A. L. (May 29, '09) Chief Engineer, Hoskins Mfg. Co., Detroit, Mich.
MARSH, Clarence W. (Dec. 26, '08) Consult. and Chem. Eng., 201 Devonshire St., Boston, Mass.; mailing address, 15 Herrick St., Winchester, Mass.
MARSH, R. J. (Sept. 27, '16) Chemist and Asst. Supt., care of R. Wallace & Son Mfg. Co., Wallingford, Conn.
MARSHALL, Jas. G. (Sept. 2, '04) Asst. Supt. Union Carbide Co.; res., 1115 Niagara St., Niagara Falls, N. Y.
MARTENS, Paul (Aug. 25, '17) Consult. Chem., 1 Beechwood Place, Elizabeth, N. J.
MARTIN, Thos C. (Feb. 26, '10) 42 Morningside Ave., New York City.
MARVIN, Arba B., Jr. (Apr. 3, '02) care of J. C. Pennie, 35 Nassau St., New York City.
MASON, Frederic S. (July 21, '11) 92 Beekman St., New York City.
MASON, Wm. D. (Aug. 25, '11) Chief Electrician, care of Standard Oil Co., Point Richmond, Cal., Box 394, Pt. Richmond, Cal.
MASTICK, Seabury C. (Feb. 27, '09) Mastick & Lucke, 2 Rector St., New York City.
MASUJIMA, Bunjiro (Mar. 4, '05) care of K. Takebe, No. 16 Gazenbo-cho, Azabuku, Tokyo, Japan.
MATHERS, Frank C. (Feb. 6, '04) 419 N. Indiana Ave., Bloomington, Ind.
MATHEWS, Dr. John A. (May 29, '09) Pres. and Gen. Mgr., Halcomb Steel Co., Syracuse, N. Y.

MATHEWSON, E. P. (May 27, '14) Royal Bank Bldg., 8 King St., East, Toronto, Canada.
MATHIAS, D. L. (Jan. 28, '16) Metallurgist, care of Mackintosh-Hemphill & Co., 803 Manufacturers Bldg., Pittsburgh, Pa.
MATSUSHITA, Nagahisa (Aug. 25, '17) care of The Nippon Steel Tube Co., Kawasaki (near Tokyo), Japan.
MATTERN, Guy G. (Aug. 25, '16) Westinghouse Elec. & Mfg. Co., 780 Ellicott Square, Buffalo, N. Y.
MAUELEN, Frederick (June 25, '09) Baker & Co., Inc., Newark, N. J.; mailing address, 79 W. Hazelwood Ave., Rahway, N. J.
MAURAN, Max (Nov. 6, '02) Eng. and Asst. Mgr., Castner Electrolytic Alkali Co., Niagara Falls, N. Y.
MAYS, S. Warren (Apr. 22, '15) Asst. Gen'l Mgr., American Cyanamid Co., 511 Fifth Ave., New York City.
MAYWALD, Frederick J. (Apr. 3, '02) Consult. Chem., 89 Pine St., New York City.
McADAM, D. J., Jr., Ph.D. (Jan. 29, '10) Metallographist, Engineering Experiment Station, Annapolis, Md.
McBERTY, Ford H. (Aug. 25, '16) Student in E. E., Cornell Univ., Bandhu, The Knoll, Ithaca, N. Y.
McBERTY, Frank R. (Sept. 25, '09) care of Western Elec. Co., Ltd., Norfolk House, Victoria Embankment, London, W. C., England.
McCLENAHAN, J. S. (June 30, '16) Asst. Supt., Electrolytic Tank House, Chile Exploration Co., Via Antofagasta, Chuquicamata, Chile.
McCONNELL, J. Y. (Apr. 3, '02) 500 N. Broad St., Philadelphia, Pa.; res., Colwyn, Pa.
McCORMACK, Harry (June 29, '07) Associate Prof., Dept. of Chemical Engineering, Armour Inst., Chicago, Ill.
McCOY, Herbert N. (Sept. 4, '03) Consult. Chem., 6030 Kenwood Ave., Chicago, Ill.
McCULLOUGH, H. F. (Apr. 29, '11) Supt., Fuse Plants, Ordnance Dept., Bethlehem Steel Co., Bethelehem, Pa.; mailing address 29 S. 13th St., Allentown, Pa.
McDONALD, Frank (Apr. 29, '11) Supt., Electrolytic Plant, D. M. Bare Paper Co., Roaring Springs, Pa.
McDONALD, Robert A. (June 25, '10) Manager, Crescent Steel Co., Crucible Steel Co. of America, Pittsburgh, Pa.; res., 304 S. Fairmount Ave.
McINTOSH, D. (Dec. 4, '02) The University of British Columbia, Vancouver, B. C., Canada.
McKAIG, W. W. (Oct. 3, '17) Director and Mgr., McKaig Mach. Foundry and Supply Works, Cumberland, Md.
McKEE, Geo. M. (Aug. 7, '03) Donnacona, Quebec, Canada.
McKELVY, Ernest C. (Aug. 27, '09) Associate Chemist, Bureau of Standards, Washington, D. C.
McKINLEY, Joseph (May 26, '10) Power Salesman, The Allegheny County Light Co., 435 Sixth Ave., Pittsburgh, Pa.
McKNIGHT, W. A. (Apr. 22, '15) Chem. Eng., Asst. in Operating Dept., Aluminum Co. of America, Niagara Falls, N. Y.
McLAUGHLIN, Dorsey E. (May 27, '11) 904 Union Oil Bldg., Los Angeles, Cal.
McMAHON, Geo. F. (Apr. 26, '17) Electrochem. Eng., Western Electric Co., 463 West St., New York City.
McMANUS, Joseph D. (June 30, '17) Student, Mass. Inst. of Tech.; mailing address, 75 Warren Ave., Marlboro, Mass.
McMILLEN, R. H. (Apr. 26, '17) Min. Eng., 134 Virginia Ave., Aspinwall, Pa.
McMURTRIE, D. H. (May 2, '17) Chem. Eng., Bureau of Mines Gas Investigation, Washington, D. C.; mailing address, 36 Lawn Ave., Woodfords, Portland, Me.
McNEILL, Ralph (Feb. 5, '03) 79 Orange St., Newark, N. J.
McNIFF, Gilbert P. (June 25, '10) Metallurgical Eng., National Tube Co., 1715 Frick Bldg., Pittsburgh, Pa.
McNITT, Robt. J. (Mar. 24, '16) Asst. Vice-Pres., The Roessler and Hasslacher Chem. Co., Perth Amboy, N. J.
McQUAID, Howard S. (June 30, '17) care of Atlas Club, Tamaqua, Pa.
MEAKER, Guy L. (July 24, '14) Pres., The Meaker Co., Chicago, Ill.; mailing address, 311 Campbell St., Joliet, Ill.
MEARS, Brainerd (Apr. 26, '17) Prof. of Chem., Thompson Chem. Labs., Williams College, Williamstown, Mass.
MEDBURY, C. F. (Oct. 22, '15) Mgr. Montreal Office, Canadian Westinghouse Co., Montreal, Quebec, Canada.
MEGROOT, J. P. (Nov. 24, '16) Electrochem. Eng., 2127 W. 48th St., Cleveland, Ohio.
MEINEKE, Otto H. (Sept. 27, '16) Construction Electrician, 422 Park Road, Ambridge, Pa.
MEREDITH, William F. (May 29, '09) Titanium Alloy Mfg. Co., Niagara Falls, N. Y.
MEREEN, John D. (May 2, '17) Elec. and Mech. Eng.. C. A. Smith Lab. and Mfg. Co., Marshfield, Oregon.
MERRILL, Chas. W. (Feb. 27, '14) Pres., Merrill Metallurgical Co., 121 Second St., San Francisco, Cal.
MERRILL, Geo. S. (Apr. 26, '17) Asst. to Chief Eng., National Lamp Works; mailing address, Nela Park, Cleveland, Ohio.

MERRILL, Millard W. (Apr. 26, '13) Electrolytic Tank House, care of Chile Copper Co., Chuquicamata, Chile, South America.
MERSHON, Ralph D. (July 1, '05) Consulting Engr., 65 W. 54th St., New York City.
MERZ, Chas. H. (Apr. 3, '02) Consult. Eng., 32 Victoria St., Westminster, London, S. W.; Collingswood Bldgs., Newcastle-upon-Tyne, England.
MERZBACHER, Aaron (June 25, '09) Chief Chem. and Met., Steel Works Dept., Henry Disston & Sons, Philadelphia, Pa.; mailing address, 6622 Torresdale Ave., Tacony, Philadelphia, Pa.
METSON, W. H. (May 25, '17) 2398 Broadway St., San Francisco, Cal.
METZ, Gustave P. (Apr. 6, '11) Supt., Consolidated Color and Chemical Co., Lister Ave. and Brown St., Newark, N. J.; res., 95 Elm St., Montclair, N. J.
METZ, H. A. (Apr. 3, '02) 122 Hudson St., New York City.
MEYER, John (Oct. 7, '05) Asst. Mgr. Sales Dept., Philadelphia Elec. Co., 1000 Chestnut St., Philadelphia, Pa.; res., 6420 Woodland Ave.
MEYER, Richard (Aug. 25, '16) Foreman, The Massillon Aluminum Co., 532 Cherry Ave., S. E., Canton, Ohio.
MEYERS, Herbert H. (June 26, '14) Res. Chemist, Mellon Inst. for Industrial Res., Pittsburgh, Pa.
MILLER, Alvin A. (Feb. 24, '17) Mgr. Railway and Lighting Division, Westinghouse Elec. and Mfg. Co., 1400 Alaska Bldg., Seattle, Wash.
MILLER, Daniel (Feb. 25, '16) Asst. Chief Chemist, Barber Asphalt Co.; mailing address, 35 Bryant Terrace, Rahway, N. J.
MILLER, Dwight D. (Oct. 26, '17) Eng., The Society for Electrical Development, 29 W. 39th St., New York City.
MILLER, Levi B. (Jan. 29, '09) El. Chemist, General Electric Co., Harrison, N. J.; mailing address, 188 Washington St., Lynn, Mass.
MILLER, L. F. (Aug. 22, '13) Room 18, Dept. of Physics, Univ. of Minnesota, Minneapolis, Minn.
MILLER, Walter (Jan. 26, '17) Process Supt., Tidewater Oil Co.; mailing address, 1624 South Madison Blvd., Tulsa, Okla.
MILLER, Dr. W. Lash (Apr. 3, '02) 50 St. Albans St., Toronto, Canada.
MILLS, J. E., Ph.D. (Apr. 16, '03) Science Hall, University of South Carolina, Columbia, S. C.
MINDELEFF, Chas. (Aug. 26, '10) Chief Chemist and Assayer, American Smelting and Refining Co., Maurer, N. J.; res., Perth Amboy, N. J.
MINER, H. S. (May 1, '07) Chief Chemist, Welsbach Light Co., Gloucester City, N. J.
MITMAN, Wm. T. (May 2, '17) Student, Lehigh Univ.; mailing address, 401 W. Broad St., Bethlehem, Pa.
MIYASAKI, Seiichi (Nov. 24, '16) 1698 Post St., San Francisco, Cal.
MOERK, Frank N. (Apr. 22, '15) Chem. Eng., 4729 N. 15th St., Philadelphia, Pa.
MOFFAT, Jas. W. (Apr. 29. '11) President, Moffat-Irving Electric Smelters, Ltd., 366 Sackville St., Toronto, Canada.
MOHN, Arnold (Aug. 25, '16) Electrochemist, 34 E. 22d St., New York City.
MOHR, Louis (Oct. 29, '10) Pres., John Mohr & Sons, 349 West Illinois St., Chicago, Ill.
MOLDENKE, Richard (Jan. 29, '09) Consult. Metallurgist, Watchung, N. J.
MOLTKEHANSEN, Ivar J. (Jan. 6, '03) Gen. Mgr., Frederiksstad Electrochemiske Fabriker, Frederiksstad, Norway.
MOODY, Dr. Herbert R. (June 29, '07) College of the City of New York, Chemical Bldg., New York City; res., 330 Convent Ave.
MOORE, Hugh K. (Feb. 25, '16) Chem. Eng., Brown Co., 93 Prospect St., Berlin, N. H.
MOORE, J. R. (Jan. 28, '16) Electrometallurgist, Oakmont, Pa.
MOORE, Wm. C. (Feb. 27, '14) Res. Chemist, National Carbon Co., Cleveland, Ohio; mailing address, 1581 Clarence Ave., Lakewood, Ohio.
MOORE, W. E. (Sept. 27, '16) Pres., W. E. Moore & Co., Cons. Engrs., 706 Union Bank Bldg., Pittsburgh, Pa.
MORDEN, Dr. G. W. (Feb. 27, '09) Manitoba Agricultural College, Manitoba, Winnipeg, Canada.
MOREHEAD, J. M. (Feb. 5, '03) Engineer of Tests, Peoples Gas Bldg., Chicago, Ill.
MORELAND, Watt L. (Apr. 26, '16) Gen. Mgr., Moreland Motor Truck Co., 1701 N. Main St., Los Angeles, Cal.
MOREY, S. R. (Apr. 29, '11) care of Mr. F. M. Waltz, Jr., 4241 Broadway, New York City.
MORGAN, Harry J. (Sept. 27, '16) Metallurgist, care of Big Indian Co., La Sal, Utah.
MORGAN, Dr. J. L. R. (Apr. 3, '02) Columbia Univ., New York City.
MORGAN, Leonard P. (Aug. 29, '08) 2003 Pine St., Philadelphia, Pa.
MORIN, Henry A. (June 30, '17) Mgr., Nicu Steel Co., Sudbury, Ont., Canada.
MORITZ, C. H. (Apr. 4, '03) care of Aluminum Co. of America, Niagara Falls, N. Y.
MORRIS, Albert W. (Jan. 26, '17) Chief Eng., Harley Co. & Morris Engineering Co.; mailing address, 54 Buckingham St., Springfield, Mass.
MORRISON, Geo. O. (June 26, '14) Metallurgist, 324 Guardian Bldg., Cleveland, Ohio.

MORRISON, M. E. (June 1, '15) Asst. Met., Standard Chem. Co., 145 N. Franklin St,. Washington, Pa.
MORRISON, Walter L. (Feb. 26, '10) Metallurgist, Box 1220, Los Angeles, Cal.
MORROW, John T. (Feb. 27, '09) care of Factory Products Export Corp., 61 Broadway, New York City.
MORSE, Willard S. (Jan. 29, '09) Dir. and Mem. Ex. Comm., Am. Smelting and Ref. Co., 120 Broadway, New York City.
MORSE, Willard V. (Dec. 30, '17) Supt. of Copper Ref., Tacoma Smelt. Co., Tacoma, Washington.
MORTIMER, James D. (Dec. 30, '16) Pres., The North American Co., 30 Broad St., New York City.
MOTT, W. R. (Mar. 5, '03) Nat. Carbon Co., Cleveland, Ohio; mailing address, 1586 Cohasset Ave., Lakewood, Ohio.
MOTTINGER, B. T. (Apr. 24, '14) Gen. Supt. Power, Ft. D. D. M. & So. R. R., Boone, Iowa; mailing address, 216 Story St.
MOULTON, Seth A. (Apr. 29, '11) Pres., Moulton Eng. Corp., 534 Congress St., Portland, Me.
MOYER, Grant C. (Jan. 29, '09) Lab. Asst., FitzGerald Labs., Niagara Falls, N. Y.
MUENCH, Reinbold K. (Nov. 24, '16) P. O. Box 293, Goldfield, Colo.
MUESER, Emil (Feb. 25, '16) Supt. Wks. No. 1, Aluminum Co. of America; mailing address, 170 Buffalo Ave., Niagara Falls, N. Y.
MUIR, J. Malcolm (Mar. 26, '10) Mgr., Metallurgical and Chemical Engineering, McGraw-Hill Publishing Co., 10th Ave. and 36th St., New York City.
MULLIGAN, J. J. (Oct. 23, '14) Met. U. S. Metals Ref. Co., East Chicago, Ind.
MURAHASHI, Sokichi (Nov. 24, '11) Met. Eng., Sokichi Murahashi Kawara Mura, near Kobe, Japan.
MURPHY, Edwin J. (Oct. 2, '02) 38 Ray St., R. D. No. 1, Schenectady, N. Y.
MURPHY, Dr. Robt. K. (May 22, '14) Technical College, Sydney, Australia.
MURRAY, Benjamin L. (Nov. 27, '09) Chemist, Merck & Co., Rahway, N. J.; mailing address, 148 Bryant St.
MUSCHENHEIM, Frederick A. (Nov. 21, '08) Vice-Pres., Hotel Astor, New York City; res., 218 W. 45th St.
MYERS, Wm. S. (Feb. 25, '16) Director Chilean Nitrate Committee, 25 Madison Ave., New York City.
NAGEL, Wm. G. (Jan. 26, '17) Pres. and Mgr., W. G. Nagel Elec. Co.; mailing address, 28 St. Clair St., Toledo, Ohio.
NAGELVOORT, Adriaan (Oct. 22, '15) Chemist, 52 E. 41st St., New York City.
NAKAMURA, Yushichiro (Nov. 23, '17) Res. Chem., The Nippon Oil Co., Ltd.; mailing address, Nippon Oil Co., Kashiwazaki, Echigo, Japan.
NAKASAWA, Yoshio (July 27, '17) Prof., Dept. of Tech., Imperial-Kioto Univ.; mailing address, Demachi-Masugata, Kioto, Japan.
NAKASHIMA, Shigemaro (Nov. 23, '17) Eng., Japan Chem. Industry, Hirota, Kanumagun, Fukushima-ken, Japan.
NAMBA, M. (Nov. 6, '03) Prof. of Elec. Eng., Kyoto Imp. Univ., Kyoto, Japan.
NASH, Clarence A. (Jan. 25, '13) Associate in Chemistry, Univ. of Chicago; mailing address, 6241 Ingleside Ave., Chicago, Ill.
NATHAN, A. F. (Mar. 27, '14) Patent Lawyer, Liberty Tower, 55 Liberty St., New York City.
NEAL, John R. H. (Apr. 26, '17) Member of Firm, Neal & Co.; mailing address, 685 Front Ave., Buffalo, N. Y.
NEEDHAM, Harry H. (Mar. 26, '15) Engineer, General Elec. Co., Harrison, N. J.; mailing address, 48 Telford St., East Orange, N. J.
NEES, A. R. (Oct. 22, '15) Chemist, Nat. Lead Co., Brooklyn, N. Y.; mailing address, 129 York St.
NESTLER, G. A. (Nov. 24, '16) Mill Foreman, Baker Mine Co., Cornucopia, Oregon.
NESTOR, John F. (Apr. 26, '17) Plater, Gen. Elec. Co.; mailing address, 31 Waterhill St., Lynn, Mass.
NEUHAUS, R. (Nov. 26, '15) Works Mgr., Electric Bleaching Co., Niagara Falls, N. Y.; mailing address, Niagara Club.
NEUMANN, Wilhelm (Aug. 28, '14) Chemist, Gullspang Elektrokemiske, A. B., Gullspang, Sweden.
NEWKIRK, Edgar D. (June 30, '17) Treasurer, care of Onondago Steel Co., Inc., Syracuse, N. Y.
NICHOLS, Wm. H., D.Sc., LL.D. (Mar. 3, '06) Pres., Nichols Copper Co., General Chem. Co., 25 Broad St., New York; res., 355 Clinton Ave., Brooklyn, N. Y.
NICHOLS, W. Standish (Apr. 3, '02) Consulting Engineer, 100 Broadway, New York City.
NICKERSON, William E. (May 29, '09) Consulting Mechanical Engineer, Gillette Safety Razor Co., So. Boston, Mass.; mailing address, 1722 Massachusetts Ave., Cambridge, Mass.
NISHIDA, Hirotaro (Oct. 26, '17) 64 Takehaya-cho, Koishikawa, Tokyo, Japan.
NISHIKAWA, Kikei (Jan. 29, '10) care of Koike, 8 Kaguraoka, Yoshida St., Kyoto, Japan.
NISWONGER, E. E. (Apr. 29, '11) President, The National Laundry Machinery Co., 731 W. Third St., Dayton, Ohio.
NOGEL, Wm. G. (Jan. 26, '17) Pres. and Mgr., The W. G. Nogel Elec. Co.; mailing address, 28-32 St. Clair St., Toledo, Ohio.

NOHARA, Dr. Tsuneo (Apr. 6, '11) Engr., 2, Iida-machi 4 Chome, Kojimachi-Ku, Tokyo, Japan.
NORMAN, Geo. M. (Apr. 3, '02) Hercules Powder Co., Wilmington, Del.
NORTHRUP, Edwin F. (Oct. 17, '07) Palmer Physical Laboratory, Princeton, N. J.
NOYES, Harry L. (Feb. 25, '16) Chief Eng., Union Carbide Co., Niagara Falls, N. Y.
NUTTING. E. G. (Dec. 30, '16) Chemist, The Carborundum Co.; mailing address, 1511 Pine Ave., No. 6, Niagara Falls, N. Y.
OAKDEN, William E. (Oct. 29, '10) 11 Lonsdale Road, Barnes, London, S. W., England.
OBER, Julius E. (Apr. 24, '09) care of West Penn Steel Co., Brackenridge, Pa.
OFFUTT, John W. (June 27, '13) Supt., Shelby Steel Co., Ellwood City, Pa.
OGDEN, John (Sept. 27, '16) Mgr., Ogden Laboratories, 230 Chancellor St., Philadelphia, Pa.
OHLWILER, Clarence H. (Apr. 6, '11) Chemist, Aetna Chem. Co., 303 Reamer Ave., Carnegie, Pa.
OLAUSSON, K. O. Ernfrid (Oct. 28, '09) Consult. Electrometallurgical Engineer, Aktiebolaget Saxberget, Trollhättan, Sweden.
OLDFIELD, Lee W. (Feb. 24, '17) Pres. and Eng., Oldfield Motors Corp.; mailing address, 506-7 Sellwood Bldg., Duluth, Minn.
OLDRIGHT, G. L. (June 30, '16) Shift Supt., Leaching Plant, care of Chile Exploration Co., Via Antofagasta, Chuquicamata, Chile.
OLSEN, T. S. (Nov. 26, '15) Det Norske A/S for Electrokemisk Industri, Bygdo Alle III, Kristiana, Norway.
OLSON, Carl G. (Aug. 25, '16) Chief Eng., Illinois Tool Works; mailing address, 154 E. Erie St., Chicago, Ill.
OLSSON, Henning (July 26, '12) Chief Designer, Hertzia, Gottenborg, Sweden.
O'NEIL, Robert Dey (Aug. 25, '17) Supt., Republic Chem. Co., P. O. Box 946, Pittsburgh, Pa.
ORDWAY, Daniel L. (May 5, '10) Asst. Director, Research Laboratory, National Carbon Co., Cleveland, Ohio; res., 1428 Ridgewood Ave., Lakewood, Ohio.
ORNSTEIN, Geo., Ph.D. (May 29, '09) Consult. and Res. Chem.; mailing address, 52 E. 41st St., New York City.
ORR, Chester A. (Jan. 28, '16) Mgr., The Cleveland Metal Products Co., Cleveland, Ohio.
OSBORNE, Loyall A. (Apr. 3, '02) Vice-Pres., Westinghouse Elec. and Mfg. Co., 165 Broadway, New York City.
OSBORNE, Sidney E. (Jan. 28, '16) Asst. Eng., Hooker Electrochemical Co.; mailing address, 170 Buffalo Ave., Niagara Falls, N. Y.
OSHIMA, Yoshikiyo, Dr. Ing. (July 30, '09) care of Mrs. Fudger, 551 W. 172d St., New York City.
OSTHEIMER, John W. (June 25, '09) Ingenieur des Arts and Manufactures, 3 rue Rabelais, Paris, France.
OWENS, Ernest W. (Feb. 24, '17) Chem., Pittsburgh Steel Co.; mailing address, 1161 Maple Ave., Monessen, Pa.
PAGE, George S. (May 29, '09) Asst. Mgr., Park Works, Crucible Steel Co. of America, Pittsburgh, Pa.
PALMER, Chas. S. (Oct. 26, '17) Fellow, Mellon Inst., Pittsburgh, Pa.
PALMER, Wm. R. (May 29, '09) 482 Farmington Ave., Hartford, Conn.
PARISH, Ralph R. (Oct. 24, '13) Rome Brass & Copper Co., Rome, N. Y.
PARKER, James H. (Nov. 26, '10) Metallurgist, Carpenter Steel Co., Reading, Pa.; mailing address, Wyomissing, Pa.
PARKHURST, C. W. (Sept. 26, '08) Supt. El. Dept., Cambria Steel Co., Johnstown, Pa.
PARKHURST, I. P. (Oct. 21, '16) Chem., Empire Gas and Fuel Co., Bartlesville, Okla.; mailing address, Box 1023.
PARKINSON, J. Carl (Feb. 25, '11) 704 Third Ave., Tarentum, Pa.
PARKS, R. E. (May 22, '14) care of Aluminum Co. of America, Maryville, Tenn.
PARMELEE, Howard C. (June 28, '12) Acting Editor, Met. and Chem. Engineering, McGraw-Hill Co., Bldg., 10th Ave. and 36th St., New York City.
PARR, Samuel W. (Jan. 7, '05) Prof. of Applied Chem., Univ. of Ill., Urbania, Ill.
PARSONS, Charles L. (Oct. 29, '08) Prof. of Inorganic Chemistry, Box 505, Washington, D. C.
PARSONS, Louis A., Ph.D. (Apr. 3, '02) Prof. of Physics, Pennsylvania College, Gettysburg, Pa.
PASCOE, C. F. (Nov. 24, '11) Metallurgist, care of Thos. Davidson Mfg. Co., Ltd., Montreal, Canada.
PATCH, N. K. B. (Jan. 23, '14) Factory Engineer, Lumen Bearing Co., Buffalo, N. Y.
PATRICK, Ralph C. (Sept. 27, '16) Chem. Eng., care of Ault & Wiborg Co., Cincinnati, Ohio; mailing address, 710 York St., Newport, Ky.
PATTERSON, C. Thos. (Oct. 3, '17) Metallurgist, Standard Chem. Co., Houston, Pa.
PATTERSON, T. A. (Sept. 27, '16) Supt., Elizabeth Plant, Morris Herrmann & Co., 3d and Trumbull Sts., Elizabeth, N. J.
PATTON, D. C. (Oct. 22, '15) Works Representative, Tolhurst Machine Works, Troy, N. Y.
PAUL, Henry N., Jr. (Apr. 3, '02) 1815 Land Title Bldg., Philadelphia, Pa.

PAULSSON, Axel (June 30, '17) Met. Eng., care of Hamilton & Hansell, 1419 Park Row Bldg., 13-21 Park Row, New York City.
PEARCE, J. Newton (Apr. 29, '11) Assistant Professor of Chemistry, The State University of Iowa, Iowa City, Iowa; res., 714 Iowa Ave.
PECK, Eugene C. (May 5, '10) Gen. Supt., The Cleveland Twist Drill Co., Cleveland, Ohio; res., 6719 Euclid Ave.
PEDDER, John (Apr. 27, '12) Chemist, West Virginia Pulp & Paper Co., Luke, Md.
PEDERSEN, Arthur Z. (Aug. 25, '16) Chemist, Edison Storage Battery Co., 86 Mt. Pleasant Ave., West Orange, N. J.
PEILER, Karl E. (Apr. 27, '12) Mech. Engineer, W. A. Lorenz, Hartford, Conn.; res., 56 Allen Place, Hartford, Conn.
PEIRCE, Wm. H. (Apr. 6, '11) Vice-President and General Mgr., Baltimore Copper Smelt. and Roll. Co., P. O. Station J, Baltimore, Md.
PENNIE, John C. (May 29, '09) Attorney at Law and Patent Solicitor, 35 Nassau St., New York City.
PENNOCK, John D., A.B., (Apr. 2, '04) Chief Chemist, Solvay Process Co., Syracuse, N. Y.
PERLEY, Geo. A. (Apr. 26, '13) Asst. Prof. of Physical Chemistry, N. H. College, Durham, N. H.
PETERS, Frazier F. (Oct. 21, '16) The Dorr Co., 227 West 90th St., New York City.
PETERSON, Andrew P. (Oct. 29, '10) Western Electric Co., Chicago, Ill.
PETINOT, Napoleon (Apr. 24, '09) Electrometallurgical Engr., care of U. S. Alloys Corp., 30 East 42d St., New York City.
PETTEE, C. L .W. (Aug. 25, '16) Owner and Mgr., The Hartford Laboratory Co., P. O. Drawer 9, Hartford, Conn.
PFANSTIEHL, Carl (Sept. 27, '16) Pres. Pfanstiehl Co., Wood Path, Highland Park, Ill.
PHILIPPS, Herbert (Nov. 6, '03) Roessler & Hasslacher Chem. Co.; res., 178 Market St., Perth Amboy, N. J.
PHILLIPS, Ross (Nov. 6, '03) Mgr., Western Alkali Ref. Co., E. Omaha, Neb.; mailing address, 3416 Sherman Ave.
PICKENS, Rufus H. (Jan. 27, '12) Southern Public Utilities Co., Clemmons, N. C.
PICKERING, Oscar W. (Oct. 2, '02) 76 Bennett Ave., Arlington, N. J.
PICKLER, A. Henry (June 1, '07) Üllös-ut 30, Budapest, Hungary.
PIERCE, James B., Jr. (May 25, '17) Chief Chem., Rollin Chem. Co., Inc.; mailing address, P. O. Box 932, Charleston, W. Va.
PIKE, Robt. D. (Dec. 30, '16) Chem. Eng., 22 Battery St., San Francisco, Cal.
PINKERTON, Andrew (Apr. 3, '03) Box 427, Pittsburgh, Pa.
PLEISS, Capt. Paul (Nov. 28, '13) Capt. in Signal Corps, Aviation Section; mailing address, 1737 "H" St., N. W. Washington, D. C.
PLOCK, Albert F. (Apr. 26, '17) Pres. Pittsburgh Met. Co.,; mailing address, 818 Park Bldg., Pittsburgh, Pa.
POND, G. G., Ph.D. July 3, '02) Prof. of Chem., State College, Pa.
POPE, Chas. E. (Feb. 25, '11) President, Coal and Coke By-Products Co., 421 Wood St.,. Pittsburgh, Pa.
POPE, Ralph W. (Apr. 26, '16) Sec'y and Treas., Aluminum Plated Ware Co., 397 Market St., Newark, N. J.
PORRO, Thomas J. (Jan. 26, '17) Pharmacist and Chem., Moore Drug Co.; mailing address, 4002 N. 27th St., Tacoma, Wash.
PRATT, E. Bruce (Dec. 26, '13) 908-16 Hippodrome Bldg., Cleveland, Ohio.
PRATT, Fred. S. (Apr. 3, '02) Stone & Webster, 147 Milk St., Boston, Mass.
PRATT, H. A. (Sept. 27, '16) Mgr., Industrial Div., N. Y. Office, Westinghouse Elec. & Mfg. Co.; mailing address, 138 Debacy Avé., N. Plainfield, N. J.
PRICE, Edgar F. (July 3, '02) 42d St. Bldg., Cor. 42d and Madison Ave., New York City.
PRICE, Wm. B. (Mar. 27, '14) Chief Chemist, Scovill Mfg. Co.; mailing address, 111 Euclid Ave., Waterbury, Conn.
PRINDLE, Edwin J. (Jan. 8, '04) Prindle, Wright & Small, Patent Lawyers, The Trinity Bldg., 111 Broadway, New York City.
PRING, John N. (Nov. 3, '06) Lecturer on Electrochem., Victoria Univ., Manchester, England.
PRITCHARD, D. A. (Oct. 26, '17) Electrochemical Plant Eng., United Alkali Co.; mailing address, "The Gables," Farnworth, Widner, England.
PRITZ, L. G. (Oct. 23, '14) Supt. of Special Steels, care of Illinois Steel Co., So. Chicago, Ill.
PRITZ, Wesley B. (May 5, '10) Asst. Supt. care of The American Carbon and Battery Co., East St. Louis, Ill.
PROCTOR, Chas. H. (Apr. 29, '11) Supervisor in Plating and Finishing Dept., F. H. Lovell & Co., Arlington, N. J.; res., 270 Argyle Place.
PROSSER, H. A. (Dec. 2, '05) Director and Member of Executive Co., American Smelt. and Ref. Co., 120 Broadway, New York City.
PULMAN, Oscar S. (May 26, '10) Asst. Superintendent, National Carbon Co., Cleveland, Ohio; res., 1507 Cohasset Ave., Lakewood, Ohio.
PULSIFER, H. B. (Nov. 24, '16) Prof. of Met., School of Mines, Butte, Mont.
PUTNAM, Wm. R. (Oct. 21, '16) Sales Mgr., Utah Power & Light Co., 515 Keams Bldg., Salt Lake City, Utah.

PYNE, Francis R. (Dec. 2, '05) U. S. Metals Ref. Co., Chrome, N. J.; res., 29 Scotland Road, Elizabeth, N. J.
QUEENY, John F. (June 1, '07) President, Monsanto Chem. Co., 1800 S. 2d St., St. Louis, Mo.
QUENEAU, A L. J. (May 1, '06) Jemeppe s/Meuse, Belgium.
QUINAN, Kenneth B. (Jan. 8, '04) Chief Chemist. De Beer's Explosive Works, Dynamite Factory, Somerset West, Cape Colony, Africa.
RAEDER, Bjorn (Jan. 23, '14) Chief of Experimental Dept., Comp. de Meteaux, Overpelt-Lammel, Neerpelt, Belgium.
RAETH, Frederick C. (Dec. 29, '11) Teacher of Electrochemistry, School of Practical Electricity, Stroh Bldg., Milwaukee, Wis.
RAIMONDO, Sebastiano (June 30, '17) 80 Maiden Lane, Room 521, New York City.
RALSTON, Oliver C. (July 23, '15) Met., Hooker Electrochemical Co., Niagara Falls, N. Y.
RAMAGE, A. S. (May 6, '05) International Color and Chemical Co., Inc., 435 Guoin St., Detroit, Mich.
RAMSEY, F. H. (Jan. 23, '14) Babcock & Wilcox Co., Bayonne, N. J.
RANDALL, A. G. (Sept. 27, '16) Gen. Foreman, Betts Plant, American Smelting and Ref Co.; mailing address, 4256 Wirt St., Omaha, Neb.
RANDALL, Henry E., Jr. (Oct. 3, '17) Power Sales Eng., Shawinigan Water and Power Co., 604 Power Bldg., Montreal, Canada.
RANDALL, J. W. H. (Jan. 29, '09) Chem. Eng., West. Va. Pulp & Paper Co., Piedmont, W. Va.
RANDALL, Merle (Nov. 27, '09) Asst. Prof. of Chem., Chemistry Bldg., Univ. of Cal., Berkeley, Cal.
RANDOLPH, Edward (Feb. 27, '09) President, Balbach Smelting and Refining Co., Bretton Hall, New York City.
RANKIN, Herbert E. (Nov. 24, '11) Cherry Hill, Albany, N. Y.
RAUTH, John W. (Sept. 27, '16) Prof. of Chem., Mt. St. Mary's College, Emmitsburg, Md.
RAY, Lieut. Horatio C. (June 27, '13) mailing address, 5853 Douglas Ave., Pittsburgh, Pa.
REBER, Col. Samuel, U. S. A. (Apr. 3, '02) Army Bldg., 39 Whitehall St., New York City.
REDFIELD, C. S. (Apr. 26, '16) Chief Chem., Ajax Rubber Co., Inc., Box 502, Trenton, N. J.
REED, Avery H. (Apr. 6, '11) Supt. of Mines, Rosiclare Lead and Fluorspar Mines, Marion, Ky.
REED, C. J. (Apr. 3, '02) 507 Brannan St., San Francisco, Cal.
REED, John C. (Apr. 29, '11) Electrical Engr., Bethlehem Steel Co., Steelton, Pa.
REED, S. Albert (June 1, '15) 113 E. 55th St., New York City.
REESE, P. P. (May 29, '09) Metallurgist, Rosslyn Farms, Carnegie, Pa.
REEVE, Amos G. (Nov. 26, '07) Research Dept., Oneida Community, Ltd., Kenwood, N. Y.
REEVE,, H. T. Mar. 24, '16) Chief Scientist, American Optical Co., Southbridge, Mass.
REICH, J. S. (Aug. 25, '16) Asst. Supt. and Chemist, Harrison Chemical Co.; mailing address, 681 Lincoln Place, Brooklyn, N. Y.
REID, John T. (Mar. 27, '09) Mining Engineer, Nevada Mining Co., Lovelock, Humboldt Co., Nev.
REID, R. R. (May 25, '17) with Grasselli Chem. Co., 1496 Arthur Ave., Lakewood, Ohio.
REINBOLD, Dr. Herman (Oct. 21, '16) Vice-Pres., Potash Reduction Co., 502 Omaha National Bank Bldg., Omaha, Neb.
REISENEGGER, Dr. Herman (June 6, '03) Charlottenburg technische Hochschule, 172 Berliner St., Charlottenburg, Germany.
REIST, Henry G. (Sept. 26, '08) Desig. Eng., Gen. El. Co., Schenectady, N. Y.
RENTON, W. L. (Oct. 26, '17) Vice-Pres. and Wks. Mgr., Electric Hal. & Metals, Welland, Ontario, Canada.
RHODIN, B. E. F. (Nov. 26, '15) care of General Chem. Co., Res. Dept., 25 Broad St., New York City.
RICH, William J. (May 26, '10) Principal Examiner, U. S. Patent Office, Room 175, Washington, D. C.
RICHARDS, Edgar (Feb. 27, '09) 60 Ayrault St., Newport, R. I.
RICHARDS, Jos. W., Ph.D. (Apr. 3, '02) Prof. of Metallurgy, Lehigh University, Bethlehem, Pa.; res., University Park.
RICHARDS, Percy J. (Nov. 24, '16) Chemist, with J. W. Richards, Assayer and Chemist, 1118 19th St., Denver, Colo.
RICHARDS, Dr. T. W. (June 3, '05) Prof. of Chem., W. Gibbs Mem. Lab., Harvard Univ., Cambridge, Mass.
RICHARDSON, C. N. (Dec. 30, '16) Private, Sanitary Corps, N. A., American Univ. Expt. Station, Washington, D. C.; mailing address, 2709 Wisconsin Ave., Washington, D. C.
RICHARDSON, E. A. (Apr. 6, '11) Chemist, 14509 Savannah Ave., East Cleveland, Ohio.
RICHARDSON, E. H. (Sept. 27, '16) Secretary, Edison Elec. Appliance Co., Ontario, Cal.

RICHARDSON, Henry K. (May 29, '09) 105 Clowes Terrace, Waterbury, Conn.
RICHARDSON, L. T. (Apr. 6, '11) Chem. Eng., care of The Cutler-Hammer Mfg. Co., Milwaukee, Wis.
RICHARDSON, W. D. (Sept. 24, '10) Chief Chemist and Chem. Eng., Swift & Co., Chicago, Ill.; res., 4215 Prairie Ave.
RICHTER, Geo. A. (Feb. 25, '16) Res. Chem. Eng., Research Lab., Brown Co., Berlin, N. H.
RICKETTS, Louis D (Apr. 24, '14) Consulting Engineer, 42 Broadway, New York City.
RIGLANDER, Moses M. (Apr. 6, '11) President, Multiple Unit Electric Co., New York City; mailing address, 47 Maiden Lane.
RIKER, John J. (Mar. 5, '03) Treas., Oldbury Electrochem. Co., 19 Cedar St., New York City.
RILEY, L. A., 2d (Aug. 25, '16) Practicing Eng., Room 607, Terminal Bldg., 103 Park Ave., New York City.
RIPPEL, Ernest G. (Oct. 23, '14) Sales Manager, Buffalo Foundry and Machine Co., 941 West Ave., Buffalo, N. Y.
RITTENHOUSE, Edward (Aug. 25, '16) Chemist, 2561 N. Front St., Philadelphia, Pa.
ROBB, Chas. D. (Nov. 27, '09) Essex Falls, New Jersey.
ROBERTS, G. I. (Feb. 24, '17) 930 St. Nicholas Ave., New York City.
ROBERTS, Isaiah L. (Oct. 2, '02) Nueva Gerona, Isle of Pines, Cuba.
ROBINSON, Almon (Apr. 3, '02) Webster Road, Lewiston, Me.
ROBINSON, Frederic W. (May 25, '12) Chemist, The Hanovia Chem. & Mfg. Co., Newark, N. J.; res., 1011 Broad St.
RODMAN, Hugh (Apr. 3, '02) care of Rodman Chem. Co., E. Pittsburgh, Pa.
ROESSLER, E. B. (Sept. 27, '16) Student in Chem. Engineering, Univ. of Cincinnati; mailing address, 3459 Brookline Ave.
ROESSLER, Dr. Franz (July 31, '07) Vice-Pres. and Supt., Roessler & Hasslacher Chem. Co., Perth Amboy, N. J.; res., 39 High St.
ROLLER, F. W. (Apr. 3, '02) Elec. Instruments (Machado & Roller), 203 Broadway, New York City.
ROLL-HANSEN, Cay (Sept. 27, '16) Supt. Keokuk Electro Metals Co., Keokuk, Iowa; mailing address, 317 Concert St.
ROLLIN, Hugh (Mar. 24, '16) Pres., Rollin Chem. Co., Inc., Charleston, W. Va.
ROMANELLI, Emilio (Jan. 28, '11) 21 Willard Ave., Bloomfield, N. J.
ROSENGARTEN, Geo. D. (Apr. 29, '11) Vice-President, Powers-Weightman-Rosengarten Co., P. O. Box 1625, Philadelphia; res., Malvern, Pa.
ROSSI, A. J. (Apr. 3, '02) Box 745, Niagara Falls, N. Y.
ROSSI, Dottor Carlo (Mar. 23, '12) Legnano (Milan), Italy.
ROSSI, Louis M. (Jan. 29, '10) Chemist, Wks. Mgr., General Bakelite Co., New York City; res., 135 Rector St., Perth Amboy, N. J.
ROTH, Charles F. (Oct. 22, '15) Mgr. National Exposition of Chem. Industries, Grand Central Palace, New York City; mailing address, 609 West 178th St.
ROUSE, Edwin W., Jr. (Feb. 27, '09) Asst. Supt., Baltimore Copper Smelting and Rolling Co., Baltimore, Md.
ROUSH, G. A. (Feb. 6, '04) Asst. Prof., Dept. of Metallurgy, Lehigh University, Editor of "Mineral Industry," Bethlehem, Pa.
ROWAND, Lewis G. (April 3, '02) care of New Jersey Zinc Co., 55 Wall St., New York City.
ROWELL, J. C. (Jan. 25, '13) Librarian, University Library, Berkeley, Cal.
ROWLAND, J. M. (Nov. 26, '15) Construction Eng., Hooker Electrochemical Co.; mailing address, 531 Buffalo Ave., Niagara Falls, N. Y.
ROWLANDS, Thos. (Feb. 27, '09) Windsor Works, North Church St., Sheffield, England.
RUBY, Chas. E. (Nov. 24, '16) 403 E. Chestnut St., Louisville, Ky.
RUFFNER, Chas. S. (Jan. 26, '17) Vice-Pres., Union Elec. Light and Power Co., 12th and Locust Sts., St. Louis, Mo.
RUHL, Louis (Dec. 2, '05) Asst. Sec., The Roessler & Hasslacher Chem. Co., 100 William St., P. O. Box 1999, New York City.
RUHOFF, O. E. (Oct. 21, '16) Eng., French Battery & Carbon Co., Madison, Wis.
RUPPEL, Henry E. K. (May 29, '09) Chemist, 66 Willow St., Wollaston, Mass.
RUSHMORE, D. B. (Oct. 2, '02) Eng. Power & Min. Dept., General Electric Co., Schenectady, N. Y.
RUSSELL, Chas. J. (Apr. 3, '02) District Manager, The Phila. Electric Co., 4522 Frankford Ave., Philadelphia, Pa.
RUSSELL, Christopher A. (May 27, '11) 1914 N St., N. W., Washington, D. C.
RUTHENBURG, Marcus (Apr. 3, '02) Met. Eng., 4 Southampton Row, Holborn, London, W. C., England.
RYAN, F. C. (Feb. 25, '16) Metallurgist, U. S. Bureau of Mines, Seattle, Wash.
RYAN, Harris J. (Nov. 30, '12) Prof. of Electrical Engineering, Leland Stanford Univ., Stanford Univ. P. O., California.
SADTLER, Samuel P., LL.D. (Apr. 3, '02) Consulting Chemist, S. P. Sadtler & Son, 210 S. 13th St., Philadelphia, Pa.
SADTLER, Samuel S. (Apr. 3, '02) with S. P. Sadtler & Son, Consulting and Analytical Chemists, 210 S. 13th St., Philadelphia, Pa.

SAKLATWALLA, B. D. (May 26, '10) General Superintendent, American Vanadium Co., Bridgeville, Pa.; res., 47 McMunn Ave., Crofton, Pa.
SALISBURY, E. F. W. (Oct. 26, '17) Electrical Contractor, 49 Wellington St., East, Toronto, Canada.
SALOM, Pedro G. (Apr. 3, '02) Commonwealth Trust Bldg., 12th and Chestnut Sts., Philadelphia, Pa.
SAMUELS, Wm. P. (May 5, '10) 120 Smithfield St., New Castle, Pa.
SARGENT, Frank C. (Jan. 29, '09) 201 Devonshire St., Boston, Mass.
SARGENT, Geo. W. (Apr. 29, '11) Metallurgist and Chemist, Electric Reduction Co., Washington, Penna.
SARGENT, R. N. (Oct. 27, '11) Works Mgr., Plant C. E., Roessler & Hasslacher Chem. Co., St. Albans, W. Va; res.. 153 State St., Perth Amboy, N. J.
SAUNDERS, Prof. A. P. (Apr. 3, '02) Hamilton College, Clinton, N. Y.
SAUNDERS, Lewis E. (Dec. 26, '07) Mgr., Abrasive Plants, Norton Co., 114 Buffalo Ave., Niagara Falls, N. Y.
SAUNDERS, Walter M. (Mar. 27, '09) Saunders & Franklin, Providence, R. I.; mailing address, 20 Dewey St.
SCHAAF, Downs (June 30, '17) Metallurgist, Buckeye Steel Castings Co., South Parsons Ave., Columbus, Ohio.
SCHABACKER, H. Eric (Oct. 26, '17) Res. Chem., Carborundum Co., Niagara Falls, N. Y.
SCHALL, Dr. C. (Aug. 29, '08) Prof. of Chemistry, Solomonstr. 1, Leipzig, Germany.
SCHAMBERG, Meyer (Dec. 27, '07) Chemist and Min. Engr., 1841 N. 17th St., Philadelphia, Pa.
SCHAPIRO, H. (Oct. 22, '15) Chief Chemist, The Ohio Match Co., Wadsworth, Ohio.
SCHILDHAUER, Edward (Dec. 26, '08.) El. and Mech. Eng., care of The Engineers Club, 32 W. 40th St., New York City.
SCHLEEDER, L. Bertram (Apr. 29, '11) Chemist, Globe Rubber Tire Mfg. Co., Trenton, N. J.
SCHLOSS, Joseph A. (Jan. 29, '09) Ore Buyer, 42 Broadway, New York City.
SCHLUEDERBERG, Carl G. (Feb. 2, '06) Asst. to Mgr., Supply Dept., Westinghouse El. & Mfg. Co., East Pittsburgh, Pa.; 210 N. Craig St., Pittsburgh, Pa.
SCHLUNDT, Herman (Nov. 5, '04) Prof. of Physical Chemistry, University of Missouri, Columbia, Mo.; res., 303 Hicks Ave.
SCHMELZ, Ernest M. (July 24, '14) Consult. Eng., 611 Moffat Bldg., Detroit, Mich.
SCHMIDT, Jay H. (Dec. 30, '17) Chem. Eng., Publicity Dept., Nat. Carbon Co.; mailing address, 11426 Clifton Road, Cleveland, Ohio.
SCHMIDT, Walter A. (Aug. 25, '17) Gen. Mgr., Western Precipitation Co., 1016 West Ninth St., Los Angeles, Cal.
SCHOCH, Dr. Eugene P. (Oct. 1, '04) Prof. of Chem., Univ. of Texas; res., 2212 Nucces St., Austin, Texas.
SCHOELLKOPF, Jacob F., Jr. (Dec. 31, '15) Chemist, P. O. Drawer 57, Buffalo, N. Y.
SCHOELLKOPF, Paul A. (Dec. 31, '15) Gen. Mgr., Hydraulic Power Co., Niagara Falls, N. Y.
SCHOEPF, T. H. (June 30, '16) care of Westinghouse Electric & Mfg. Co., East Pittsburgh, Pa.
SCHOLL, G. D. (May 25, '17) 929 N. 12th St., Keokuk, Iowa.
SCHRAMM, Edward (Oct. 21, '16) Asst. Chemist, Bureau of Standards, 16 W. Kirke St., Chevy Chase, Md.
SCHRANTZ, Todd L. (July 28, '16) Mgr., Supply Division, Westinghouse E. & M. Co.; mailing address, 780 Ellicott Square, Buffalo, N. Y.
SCHREY, Adolf (Nov. 26, '10) Sidonianstrasse 10, Dresden A, Germany.
SCHROEDER, C. M. Edward (May 29, '09) Chief Assistant, Standard Bleaching Co., Carlton Hill, N. J.; mailing address, 235 Wood St., Rutherford, N. J.
SCHUBERT, Bruno H. (Aug. 25, '16) Inspector and Chemist, R. W. Hunt & Co.; mailing address, 224 Jane St., Weehawken, N. J.
SCHUBERT, H. R. (Nov. 24, '16) Met. Eng., Jerpeland, Stavanger, Norway.
SCHUELER, J. L. (Sept. 27, '16) Chemist and Met., Keystone Steel and Wire Co., Peoria, Ill.
SCHUETZ, Fred. F. (Oct. 1, '04) Patent Attorney, Room 1352, 50 Church St., New York City.
SCHUELTE, Walter B. (Oct. 29, '10) Treasurer, C. F. Burgess Laboratories, 625 Williamson St., Madison, Wis.
SCHULTZ, Louis Claude (Oct. 22, '15) Chem. Eng., care of The Tungsten Products Co., 2031 12th St., Boulder, Colo.
SCHULZE, Paul (Oct. 26, '17) Res. Chem.; United Piece Dye Wks.; mailing address, 287 Union St., Hackensack, N. J.
SCHUYLER, A. H. (June 2, '16) Metallurgist, International Nickel Co., Bayonne, N. J.
SCHWARZ, Ralph C. (May 5, '10) Electrical Engineer, Taylor Instrument Co., Rochester, N. Y.
SCOTT, Professor Chas. F. (Aug. 27, '09) 284 Orange St., New Haven, Conn.
SCOTT, L. C. (Oct. 21, '16) 976 St. John's Place, Brooklyn, N. Y.
SCOTT, Wirt S. (Aug. 25, '16) Westinghouse Elec. & Mfg. Co., East Pittsburgh, Pa.; mailing address, 121 Linden Ave., Edgewood Park, Pa.
SEEDE, John A. (May 29, '09) Elec. Eng., General Electric Co., Schenectady, N. Y.; mailing address, Route 49, Schenectady, N. Y.

SERGEANT, Elliott M. (October 1, '04) Box 54, Niagara Falls, N. Y.
SERVIS, Oscar E. (Feb. 24, '17) Foreman Electroplater, Felt & Tarrant Co.; mailing address, 5305 Warner Ave., Chicago, Ill.
SETHMAN, Geo. H. (Nov. 6, '03) Engineer and Constructor, Suite 250, Equitable Bldg., Denver, Colo.
SEWARD, Geo. O. (April 3, '02) 99 Cedar St., New York City.
SEYFERT, Stanley S. (Oct. 29, '08) Assoc. Prof., El. Eng., Lehigh Univ.; res., 456 Chestnut St., Bethlehem, Pa.
SHARP, Clayton H., Ph.D., (Nov. 26, '07) Technical Director, Electrical Testing Laboratories, 80th St. and East End Ave., New York City; res., White Plains, New York.
SHARPLES, Stephen P. (Feb. 27, '09) Consulting Chemist, Campbell Magnetic Separator Co., Boston, Mass.; mailing address, 26 Broad St.
SHATTUCK, A. F. (Apr. 3, '02) Chief Chem., The Solvay Process Co., Detroit, Mich.; mailing address, 615 Canon Drive, Beverly Hills, Cal.
SHAW, E. C. (Apr. 3, '02) North Portage Path, Akron, Ohio.
SHEFFIELD, Wesley T. (Aug. 25, '17) Chief Chem., Plattsburg Alloys Corp., Plattsburg, N. Y.
SHELDON, Dr. Samuel (Apr. 3, '02) Prof. of Physics and Elec. Eng., Polytechnic Inst., Brooklyn, N. Y.
SHEPHERD, F. A. (Nov. 27, '14) Electroplating Eng., International Steel and Ordnance Co., Lowell, Mass.
SHEPHERD, F. R. (Sept. 25, '14) Chief Electrical Inspector, P. O. Box 361, Dunedin, N. Z.
SHERK, Harold C. (July 27, '17) Supt., Electrode Dept., care of Beckman & Linden Eng. Corp., 422 Balboa Bldg., San Francisco, Cal.
SHIELDS, Dr. John (Dec. 4, '02) Minas de Rio Tinto, Provincia de Huelva, Spain.
SHIELDS, James E. (June 30, '17) Asst. Chem., National Carbon Co.; mailing address, 81 Duane Ave., La Salle, N. Y.
SHIMER, Edward B. (Jan. 28, '11) Asst., Porter W. Shimer Laboratory, Paxinosa Ave., Easton, Pa.
SHINDELL, Harry F. (Jan. 26, '17) Supt., T. A. Wilson & Co., Inc., 925 McKnight St., Reading, Pa.
SHIVERICK, Myron D. (Nov. 21, '08) 25 Menands Road, Menands, N. Y.
SHOAFF, P. S. (Sept. 27, '16) Chem. Eng., Sea Products Co. & Oceanic Eng. Corp.; mailing address, 421 Daisy Ave., Long Beach, Cal.
SICARD, Hugh C. (June 30, '17) Electrometallurgical Eng., United States Alloy Corp.; mailing address, Hotel Touraine, Buffalo, N. Y.
SIEGER, Geo. N. (Apr. 27, '12) Asst. to Mgr., Electrolytic Dept., Davis-Bournonville Co., Marion Station, Jersey City, N. J.
SIEVERING, Philip (Apr. 26, '13) Electroplater, Philip Sievering Co., 54 Nairn Place, Newark, N. J.
SIMMERS, Arthur L. (May 29, '09) Eng., Hooker Electrochemical Co., Niagara Falls, N. Y.; mailing address, 19 Sugar St.
SIMPSON, Louis (May 29, '09) 172 O'Connor St., Ottawa, Canada.
SIMS, C. E. (June 1, '15) Michigan Electrochemical Co., Menominee, Mich.
SKILLMAN, Verne (Mar. 26, '10) care of General Aluminum & Brass Mfg. Co., East Grand Blvd. and St. Aubin Ave., Detroit, Mich.
SKINNER, Chas. E. (Dec. 31, '09) Engineer, Research Division, Westinghouse Elec. and Mfg. Co., Wilkinsburg, Pa.; mailing address, 1309 Singer Place.
SKINNER, H. J. (Apr. 3, '02) care of A. D. Little, Cambridge, Mass.
SKINNER, L. B. (Oct. 23, '14) V. P. and Gen. Mgr., Western Chem. Mfg. Co., Denver, Colo.
SKOWRONSKI, Stanislaus (June 1, '07) care of Raritan Copper Works, Perth Amboy, New Jersey.
SLATER, Wm. A. (Feb. 25, '11) Supt., Gulf Refining Co., P. O. Box 1024, Ft. Worth, Texas.
SLOCUM, Frank L., Ph.D. (Dec. 4, '03) Pres., Technical Service Corp., Empire Bldg., Pittsburgh, Pa.; mailing address, 401 S. Linden Ave., E. E.
SMITH, Acheson (Aug. 31, '07) Vice-Pres. and Gen. Mgr., Acheson Graphite Co.; res., 113 Jefferson Ave., Niagara Falls, N. Y.
SMITH, A. T. (Dec. 4, '03) 257 Royal Liver Bldg., Liverpool, England.
SMITH, Prof. A. W. (Apr. 3, '02) 11333 Bellflower Road, Cleveland, Ohio.
SMITH, Dyer (June 28, '12) Patent Lawyer, 32 Liberty St., New York City; mailing address, 9 Wilde Place, Montclair, N. J.
SMITH, E. A. Cappelen (May 9, '03) Consulting Engineer, 120 Broadway, New York City.
SMITH, Dr. Edgar F. (June 3, '05) Provost, Univ. of Penna., Philadelphia, Pa.
SMITH, Edmund S. (Apr. 3, '02) Chemist, The Carborundum Co., Niagara Falls, N. Y.
SMITH, E. W. (Apr. 3, '02) 74 E. Penn St., Germantown, Philadelphia, Pa.
SMITH, Frank T. (Dec. 30, '16) Chem., Chile Exploration Co., Chuquicamata, Chile.
SMITH, F. Warren (May 9, '03) Provo City, Utah.
SMITH, H. F., Jr. (Aug. 25, '16) Eng., E. W. Clark & Co., 321 Chestnut St., Philadelphia, Pa.
SMITH, Harold H. (Jan. 27, '12) Edison Storage Battery Co., Orange, N. J.
SMITH, John H. (Jan. 26, '17) Consult. Eng., 1319-21 First National Bank Bldg., Milwaukee, Wis.

SMITH, Walter C. (Oct. 23, '14) Supt. Silver Bldg., U. S. Metals Ref. Co., Chrome, N. J.
SMULL, Judson G. (Nov. 26, '07) Res. Chem., New Jersey Zinc Co. (of Penna.), Palmerton, Pa.
SMYTHE, Edwin H. (May 29, '09) Electrical Engineer, 738 Monadnock Bldg., Chicago, Ill.
SNEATH, Wm. H. (May 25, '12) Asst. Supt., Electro Metallurgical Co., La Salle, N. Y.
SNOOK, H. Clyde, A.M. (Nov. 26, '07) 224 Linden Ave., Oak Park, Ill.
SNOW, Wm. M. (Feb. 25, '11) Supt. of Acid Works, Illinois Zinc Co., Peru, Ill.
SNOWDON, R. C. (Feb. 3, '06) Operation Expert, Hooker Electrochemical Co.; mailing address, 21 C St., Niagara Falls, N. Y.
SNYDER, Chas. G. (June 30, '17) Chief Chem., Duquesne Reduction Co.; mailing address, 5212 Harriet St., Pittsburgh, Pa.
SNYDER, Fred. T. (June 6, '03) Vice-Pres., Industrial Elec. Furnace Co., Chicago, Ill.; mailing address, Room 1405, 53 W. Jackson Blvd.
SNYDER, J. L. K. (May 5, '10) Supt., Silverford Plant, Aetna Chem. Co., Inc., Newton Hamilton, Pa.
SODERBERG, Carl W. (Oct. 29, '10) Det norske Aktieselskap for Electrokemiske Industri, Christiania, Norway.
SOLVAY, Armand (Jan. 8, '04) Mgr., The Solvay Co. of Brussels, 33 rue du Prince Albert, Brussels, Belgium.
SOMDAL, John A. (Jan. 22, 1915), Res. Chemist, 1119 W. Franklin Ave., Minneapolis, Minn.
SPARRE, Fin, Dr. (Oct. 26, '12) 606 W. 20th St., Wilmington, Delaware; mailing address, Director, care of Experimental Station, E. I. du Pont de Nemours & Co., Henry Clay P. O., Delaware.
SPAULDING, Harris W. (Dec. 28, '17) Owner, National Copper Wks.; mailing address, 140 N. Washington St., Boston, Mass.
SPEED, Buckner (Dec. 31, '14) Head of Physical Lab., Western Electric Co.; mailing address, 463 West St., New York City.
SPEIDEN, Clement C. (Nov. 6, '02) 46 Cliff St., New York City.
SPEIDEN, Eben C. (Feb. 2, '06) Vice-Pres. and Gen. Mgr., care of Isco Chem. Co., Inc., Niagara Falls, N. Y.
SPELLER, Frank N. (Jan. 29, '10) Met. Eng., National Tube Co., 1824 Frick Bldg., Pittsburgh, Pa.; res., 1624 Wightman St.
SPENCER, A. G. (Sept. 27, '16) Consult. Chem. & Met., 619 Transportation Bldg., Montreal, Canada; mailing address, 4076 Tupper St., Westmount, Quebec, Can.
SPERRY, Elmer A. (Apr. 3, '02) 100 Marlborough Road, Prospect Park, South, Brooklyn, N. Y.
SPERRY, R. S. (June 1, '15) Res. Eng., Scovill Mfg. Co., Waterbury, Conn.
SPICER, C. W. (Aug. 25, '16) Vice-Pres. and Chief Eng., Spicer Mfg. Co., Plainfield, N. J.; mailing address, 417 Central Ave.
SPILSBURY, Edmund G. (Jan. 28, '11) Consulting Engr., 29 Broadway, New York City.
SPRAGUE, Edmund C. (July 31, '08) Chemist, care of E. G. Acheson, Ltd., 5 Chancery Lane, London, W. C., England.
STAFFORD, Samuel G. (May 26, '10) Vice-Pres. and Gen. Mgr., Vulcan Crucible Steel Co., Aliquippa, Pa.; res., State St., Coraopolis, Pa.
STAHL, Nicholas (Aug. 25, '16) Commercial Eng., Westinghouse El. & Mfg. Co.; mailing address, 204 Hawthorn St., Swissvale Station, Pittsburgh, Pa.
STAMPS, F. A. (June 2, '06) Chem., care of Phosphorus Compounds Co., Niagara Falls, N. Y.
STANLEY, Frederick C. (Aug. 25, '16) Editor, The Metal Record and Electroplater; mailing address, 260 John St., Bridgeport, Conn.
STANSFIELD, A., D.Sc., A.R.S.M. (Jan. 8, '03) Prof. of Metallurgy, McGill Univ., Montreal, Canada.
STARK, Edgar E. (Jan. 29, '09) City Electrician, Box 526, Christ Chruch, New Zealand.
STARK, Paul A. (July 27, '17) Res. Chem., 2655 Dwight Way, Berkeley, Cal.
STARR, Lieut. Arthur B., Jr. (Feb. 25, '16) 154 F. A. Brigade Headquarters, Camp Meade, Md.
STATHAM, Noel (Oct. 17, '07) Mgr. Chem. Dept., West Virginia Pulp & Paper Co., 200 5th Ave., New York City.
STEIN, Walter M. (Jan. 8, '03) Pres. and Gen. Mgr., Primos Chem. Co., Primos, Delaware Co., Pa.
STEINMETZ, Charles P. (Aug. 7, '02) Chief Consult. Eng., Gen. Elec. Co.; res., Wendell Ave., Schenectady, N. Y.
STEPHENS, Chas. E. (June 2, '16) Mgr., Illuminating Section, Supply Dept., Westinghouse Elec. & Mfg. Co.; mailing address, 1213 Center St., Wilkinsburg, Pa.
STEPHENSON, Frank T. F. (May 5, '10) Chemical Dept., Detroit Technical Institute, Detroit, Mich.; res., 873 Trumbull Ave., Detroit, Mich.
STEPHENSON, H. L. (July 28, '16) Salesman, Westinghouse Elec. & Mfg. Co., 780 Ellicott Square, Buffalo, N. Y.
STEVENS, J. Franklin (Sept. 26, '08) Engr., 1326 Chestnut St., Philadelphia, Pa.
STEVENS, W. L. (Sept. 15, '15) Metallurgical Accountant, Braden Copper Co., Rancagua, Chile.

STEVENSON, Reston (Apr. 22, '15) Asst. Prof. of Chemistry, The College of the City of N. Y., New York City.
STEWART, Harry M. (Sept. 27, '16) Supt. of Blast Furnaces, Carnegie Steel Co., Duquesne, Pa.
STEWART, Marshall E. (Oct. 21, '16) Works Mgr., Plant 3, Roessler & Hasslacher Chemical Co.; mailing address, 45 Woodruff Place, Perth Amboy, N. J.
STEWART, Reid T. (July 29, '10) Professor of Mechanical Engineering, University of Pittsburgh, Pittsburgh, Pa.; res., 1524 Shady Ave.
STEWART, Robert Stuart (Sept. 26, '08) Consult. Elec. Eng., 530 St. Paul Ave., Detroit, Mich.
STICHT, Robert C. (May 29, '09) General Mgr., Mt. Lyell M. & R. Co., Ltd., Queenstown, Tasmania, Australia.
STILLESEN, Job M. A. (May 25, '17) Niagara Falls, N. Y.
ST. JOHN, H. M. (Mar. 24, '16) Res. Eng., Commonwealth Edison Co.; mailing address, 28 N. Market St., Chicago, Ill.
STOCKTON-ABBOTT, Lyle (July 28, '16) Hotel Plaza, Port Arthur, Texas.
STOLL, Clarence G. (Mar. 27, '09) Tech. Supt., Western Elec. Co., Hawthorne Station, Chicago, Ill.
STONE, Chas. A. (Apr. 3, '02) Stone & Webster, 147 Milk St., Boston, Mass.
STONE, Edmund C. (Dec. 30, '17) System Operator, Duquesne Light Co., 435 6th Ave., Pittsburgh, Pa.
STONE, George W. (Mar. 27, '09) care of Hooker Electrochemical Co., Niagara Falls, N. Y.
STONE, I. F. (Jan. 29, '09) Pres., Nat. Aniline & Chemical Co., 100 William St., New York City.
STONE, Joseph P. (Nov. 21, '08) Purchasing Engineer, care of Lindetevas-Stokirs, 11 Broadway, New York City.
STONE, John Stone (Jan. 29, '09) Consult. Elec. Eng., 153 East 18th St., New York City.
STOREY, Oliver Wendell (Feb. 25, '11) Met. Eng., care of C. F. Burgess Labs., Madison, Wis.
STOTTER, Max (May 25, '17) Proprietor, care of Pennsylvania Smelt. Co., 1385 W. Jefferson Ave., Detroit, Mich.
STOUGHTON, Bradley (Dec. 31, '09) Sec'y American Institute of Mining Engineers, 29 W. 39th St., New York City.
STRACHAN, E. K. (Oct. 3, '17) Res. Chem., National Aniline & Chem. Co.; mailing address, 234 Saranac Ave., Buffalo, N. Y.
STRATTON, W. G. (Apr. 26, '17) Electroplater, R. N. Bassett Co., Derby, Conn.; mailing address, 153 Elm St., Bridgeport, Conn.
STRAUSS, Frank A. (Mar. 26, '15) 196 S. Franklin St., Wilkes-Barre, Pa.
STUPAKOFF, S. H. (Feb. 27, '14) Manufacturer, 401-431 Amberson Ave., Pittsburgh, Pa.
STYRI, Haakon (Feb. 25, '11) Asst. Prof. of Met., Carnegie Inst. of Technology, Pittsburgh, Pa.
SULLIVAN, Alan P. (June 30, '17) Terryville, Conn.
SUMAN, Robert G. (Apr. 26, '17) Electroplater, 66 Notre Dame Ave., Dayton, Ohio.
SUMMERS, Leland L. (Sept. 26, '08) L. L. Summers & Co., First Nat'l Bank Bldg., Chicago, Ill.
SWANN, Theodore (May 2, '17) Pres., Southern Manganese Corp., Brown-Marx Bldg., Birmingham, Ala.
SWARTLEY, Henry P., Jr. (June 1, '15) Sales Engineer, care of Davis Bournonville Co., Marion Station, Jersey City, N. J.
SWENARTON, W. Hastings (Apr. 27, '12) Firm of Merwin & Swenarton, 2 Rector St., New York City.
SYMMES, Whitman (Dec. 4, '03) Min. Eng., Virginia City, Nev.
TABER, Geo. H., Jr. (Dec. 30, '16) 325 W. 12th St., Tulsa, Okla.
TACHIHARA, Jin (Mar. 27, '14) Electrical Engineer, Mitsu Bishi Dockyard and Engine Wks., Kobe, Japan.
TADA, Kozo (July 28, '16) Supt. Power House, Inawashire Hydro-Elect. Power Co., care of Bandai Post Office, Fukushimaken, Japan.
TADA, Commander S. (Sept. 4, '03) Naval Inspector's Office, Kawasaki Dockyard, Kobe, Japan.
TAFEL, Theodore, Jr. (Oct. 21, '16) Standard Sanitary Mfg. Co., Pittsburgh, Pa.
TAGGART, Walter T. (Nov. 6, '02) University of Pennsylvania, Dept. of Chem., Philadelphia, Pa.
TAKAHASHI, Taketaro (July 30, '09) Shinano Elec. Co., Yoshida, Nagano, Japan.
TAKAMINE, Jokichi (Feb. 27, '09) Research Chemist, Parke, Davis & Co., 550 W. 173d St., New York City.
TAKIKAWA, Yasuo (Oct. 21, '16) Member Asso. Chem. Industry, Tokyo, Japan; mailing address, Section D, Taylor Hall, Bethlehem, Pa.
TALBOT, Prof. H. P. (Jan. 8, '04) Prof. of Inorganic Chem., Mass. Inst. of Tech.; res., 273 Otis St., West Newton, Mass.
TANNER, W. Lee (Sept. 27, '16) Chief Chemist, New Castle Plant, Bethlehem Steel Co.; mailing address, Box 249, Wilmington, Del.
TATE, John V. E. (Mar. 27, '09) Chief Line Inspector, The Cauvery Elec. Power Scheme, Kankanhallie, Mysore State, India.
TATSUMI, Eiichi (Mar. 24, '16) Chem. Eng., care of Miike Colliery, Omuda-machi, Fukuoka-ken, Japan.

TAYLOR, Dr. Chas. E. (May 29, '09) Taylor Chem. Co., Penn Yan, N. Y.
TAYLOR, Floyd D. (Dec. 31, '09) 85 Ives St., Waterbury, Conn.
TAYLOR, H. N. (Nov. 24, '16) Senior Member Firm, N. & G. Taylor Co.; mailing address, 300 Chestnut St., Philadelphia, Pa.
TAYLOR, Hugh S. (June 1, '15) Instructor in Physical Chem., Graduate College, Princeton, N. J.
TAYLOR, John B. (June 21, '11) Consulting Elec. Engr., 100 Broadway, New York City; mailing address, 23 Lowell Road, Schenectady, N. Y.
TEEPLE, Dr. J. E. (May 1, '06) Consulting and Mfg. Chemist, 50 East 41st St., New York City.
TEEPLE, Oliver J., Jr. (Feb. 26, '15) Tech. Asst., Du Pont Powder Co., 953 Du Pont Bldg.; Wilmington, Del.
TEMPLE, Sterling (Sept. 27, '16) In charge of Industrial Chemistry Courses, Univ. of Minnesota; mailing address, 1758 Blair St., St. Paul, Minn.
THARALDSEN, Filip (Apr. 29, '11) General Mgr., A/s Ilens Smelteverk, Dronningensgate 22, Kristiania, Norway.
THATCHER, C. J., Ph.D. (Jan. 7, '05) Pat. Exp., No. 1 Riverdale Ave., New York City.
THEURER, Geo. A. (May 25, '17) Met. Chem.; 286 Mills St., Buffalo, N. Y.
THOMAS, Bruno (June 28, '12) Consulting Engineer, 3909 E. Howell St., Seattle, Washington.
THOMAS, Geo. E. (June 30, '16) Chemist and Electroplater, Factory H, International Silver Co.; mailing address, 759 Broad St., Meriden, Conn.
THOMPSON, Prof. Elihu (Sept. 17, '03) Elec., Gen. Elec. Co., 22 Monument Ave., Swampscott, Mass.
THOMPSON, J. G. (Feb. 25, '16) Asst. in Chemistry, Morse Hall, Cornell Univ., Ithaca, N. Y.
THOMPSON, John (Feb. 27, '09) Civil and Mechanical Engineer, 253 Broadway, New York City.
THOMPSON, M. deK., Jr. (Nov. 6, '03) Associate Prof. of Electrochem., Mass. Inst. of Tech., Cambridge, Mass.
THOMPSON, Maurice R. (Dec. 30, '16) Electrometallurgical Eng., Gen. Elec. Co., Power and Mining Dept., Schenectady, N. Y.
THORDARSEN, Chester, H. (Apr. 6, '11) President, Thordarsen Elec. Mfg. Co., 216-220 S. Jefferson St., Chicago, Ill.; res., 1416 Leland Ave.
THORNE, C. A. (May 22, '14) Consult. Engr., Ovre Slotsgate 5, Kristiania, Norway.
THRELFALL, Richard (Apr. 4, '03) Oakhurst, Church Road, Goghaston, Birmingham, England.
THUM, Wm. (Dec. 31, '14) Supt. of Electrolytic Lead Refinery, U. S. Metals Ref. Co.; mailing address, 44 Webb St., Hammond, Ind.
THURBER, John H. (Oct. 21, '16) Chemist and Assayer, Gorham Mfg. Co.; mailing address, 210 Lexington Ave., Providence, R. I.
THURSTON, Louis S. (Oct. 3, '17) Engr. and Salesman, care of General Elec. Co., P. & M. Dept., Schenectady, N. Y.
THWING, Dr. Charles Burton (Nov. 27, '09) Pres., Thwing Instrument Co., 3339-41 Lancaster Ave., Philadelphia, Pa.
TIEMANN, Hugh P. (Oct. 24, '13) Asst. Metallurgical Eng., Carnegie Steel Co., 563 Frick Annex, Pittsburgh, Pa.
TILLBERG, Erik W. (Nov. 6, '02) Westervik, Sweden.
TILLMAN, Richard H. (Aug. 25, '16) Mgr., New Business Dept., Consolidated Gas, Electric Light and Power Co., Lexington and Liberty Sts., Baltimore, Md.
TINGBERG, Otto (July 29, '10) Editor of Jern-Kontorets Annaler, Jernkontoret, Stockholm, Sweden.
TOCH, Maximilian (Nov. 6, '03) 320 Fifth Ave., New York City.
TONE, F. J. (Apr. 3, '02) Works Mgr., The Carborundum Co., Niagara Falls, N. Y.
TOOMER, James E. (Jan. 26, '17) Chief Chem., American Zinc Co. of Tenn.; mailing address, Box 63, Mascot, Tenn.
TORELL, T. F. (Aug. 28, '14) Chief Chemist and Asst. Supt., Kristiansand Nikkelraff. verk, Kristiansand, S. Norway.
TOWER, O. F. (Dec. 31, '14) Prof. of Chemistry, Adelbert College, Cleveland, Ohio.
TOWNSEND, C. P. (Apr. 3, '02) 918 F St., N. W., Washington, D. C.
TRANTIN, Jacob, Jr. (Feb. 25, '16) Res. Met., Inspector of Ordnance, U. S. N., Goss Printing Press Co., 1535 S. Pauline St. Chicago, Ill.
TREACY, Cyril S. (Feb. 24, '17) Res. Chem., Chas. Pfiger & Co.; mailing address, 33 Press Ave., Norwood, Mass.
TREADWELL, John C. (Feb. 24, '17) Pres. and Gen. Mgr.; Dryoxide Chem. Co.; mailing address, 56-58 Pine St., New York City.
TUCKER, Samuel A. (Mar. 3, '06) Adjunct Prof. Electrochem., Columbia Univ.; res., 155 E. 61st St., New York City.
TUDHOPE, Hugh R. (Oct. 26, '17) Partner, Turnbull Electrometals, Orillia, Ontario, Canada.
TURNBULL, Robert (Feb. 27, '09) Box 416, Welland, Ont., Canada.
TURNER, M. R. (Oct. 27, '11) Manager and Chem., A/s Stangfjordens Elektromiske Fabriker, Gjorde, Stangfjorden pr. Bergen, Norway.
TURNOCK, E. Hill, Jr. (July 27, '17) Student, Carnegie Inst. of Tech.; mailing address, 5329 Beeler St., Pittsburgh, Pa.
TURNOCK, L. C. (Nov. 26, '10) Dept. of Chemical Engineering, Carnegie Institute of Technology, Pittsburgh, Pa.

TWINING, Fredericke (Feb. 24, '17) Mgr., The Twining Labs., Fresno, Cal.
TYLER, Walter S. (July 28, '16) Supt. Matrix Dept., American Graphophone Co., 503 Colorado Ave., Bridgeport, Conn.
UHLENHAUT, F., Jr. (May 26, '10) Chief Engineer, Allegheny County Light Co. and Pittsburgh Railways Co., 435 Sixth Ave., Pittsburgh, Pa.
UNDERWOOD, C. W. (Aug. 25, '16) District Mgr., Westinghouse Elec. and Mfg. Co., 780 Ellicott Square, Buffalo, N. Y.
UNGER, Geo. N. (Sept. 27, '16) Student, Purdue Univ., Box 104, West Lafayette, Ind.
UNGER, John S. (May 26, '10) Manager, Central Research Bureau, 1053-1057 Frick Annex Bldg., Pittsburgh, Pa.
UNGER, Magnus (Nov. 27, '09) Elec. Eng., The General Electric Co., Pittsfield, Mass.; res., 51 Lincoln Terrace.
UNZICKER, Samuel P. (Sept. 27, '16) Principal, High School, White Pigeon, Mich. R. F. D. to Scott, Ind.
VALENTINE, Irving R. (July 21, '11) Chemist, Fulton Steel Co., Fulton, N. Y.
VALENTINE, Wm. (Sept. 17, '03) 22 Mitchell Ave., Waterbury, Conn.
VAN ARSDALE, Geo. D. (June 21, '11) Consulting Chemist, Phelps, Dodge & Co., 99 John St., New York City.
VAN BRUNT, Chas. (Feb. 27, '09) Chemist, Research Laboratory, General Electric Co., Schenectady, N. Y.
VAN DEVENTER, Harry R. (Feb. 27, '09) Elec. Engr., Sumter Tel. Mfg. Co., Sumter, S. C.
VAN KEUREN, Wm. L. (Dec. 31, '15) Eng., Edison Lamp Works, General Electric Co.; mailing address, 78 Danielson St., North Bergen, N. J.
VAN SICKLEN, Wm. J. (Oct. 22, '15) 455 Minna St., San Francisco, Cal.
VASSAR, H. S. (Oct. 22, '15) Lab. Eng., Public Service Elec. Co., 39 Willard Ave., Bloomfield, N. J.
VAUGHN, C. F. (Nov. 6, '02) Supt. Castner Electrolytic Alkali Co., University Club, Niagara Falls, N. Y.
VIOL, Chas. H. (Dec. 31, '15) Director, Radium Res. Lab., Standard Chem. Co.; mailing address, Box 22, Oakland Station, Pittsburgh, Pa.
VOGELER, W. R. (Feb. 24, '17) Export Mgr., King Car Co.; mailing address, 86 Harrison Ave., Baldwin, Long Island, N. Y.
VOGT, Louis Fenn (Oct. 3, '17) Wks. Mgr., care of Standard Chem. Co., Canonsburg, Pa.
VOM BAUR, Carl H. (Oct. 26, '12) Consult. Eng., 30 Church St., New York City.
VON FOREGGER, R., Ph.D. (Sept. 2, '05) 31 W. 42d St., Aeolian Bldg., New York City.
VON KROGH, Johan (Dec. 30, '17) District Mgr., A/s Bjölvefossen, Sndre. Aalinth, Hardanger, Norway.
VOORHEES, Louis A. (Apr. 3, '02) 111 Carroll Place, New Brunswick, N. J.
VORCE, L. D. (July 31, '07) Gen. Supt., care of Canadian Salt Co., Windsor, Ont., Canada.
VOSS, William (June 30, '17) Foreman of Plating, Polishing and Finishing Dept., Intertype Corp.; mailing address, 2011 Palmetto St., Brooklyn, N. Y.
WADDELL, Montgomery (Mar. 27, '09) Consulting Engr., 30 Church St., New York City.
WADSWORTH, Frank L. O. (July 1, '04) 1347 Oliver Bldg., Pittsburgh, Pa.
WAGNER, Henry L., M.D., Ph.D. (Apr. 3, '02) 518 Sutter St., San Francisco, Cal.
WALDO, Wm. B. (June 21, '11) Captain, Engineers, U. S. R., France.
WALKER, Arthur L. (Feb. 27, '09) Prof. of Metallurgy, Columbia University, New York City.
WALKER, E. C., 3d (Oct. 22, '15) 191 Babcock St., Brookline, Mass.
WALKER, Geo. H. P. (Aug. 25, '16) Chief Chemist, Canadian Salt Co., Ltd.; mailing address, Box 248, Sandwich, Ontario, Canada.
WALKER, James W. (July 26, '12) Managing Director, Alexander Walker & Co., Marine Lodge, Irvine, Scotland.
WALKER, Dr. W. H. (Aug. 7, '02) Prof. Chem. Eng., Mass. Inst. of Tech., Cambridge, Mass.
WALLACE, Walter (Jan. 29, '09) Assistant Works Mgr., Oldbury El. Chem. Co., Niagara Falls, N. Y.
WALLOWER, Frank C. (Nov. 24, '16) 112 Sergeant Ave., Joplin, Mo.
WALMSLEY, Walter M. (Sept. 24, '10) Alabama Power Co., Brown-Marx Bldg., Birmingham, Ala.
WALSH, Philip C., Jr. (Apr. 7, '06) Member Walsh's Sons & Co., Metal and Machinery Merchants, 19 Grant St., Newark, N. J.
WALTER, R. J. (Sept. 27, '16) Consulting Mining and Met. Eng., Kittmas Mines Co.; mailing address, 2130 Downing St., Denver, Colo.
WALTERS, Joseph (Nov. 27, '14) Foreman Electroplater, The Southern Stove Works; mailing address, 2019 Park Ave., Richmond, Va.
WARD, A. T. (Apr. 26, '13) Experimental Eng., Bellefonte, Pa.
WARD, H. Lee (Dec. 30, '16) Instructor in Chemistry, School of Chem., Univ. of Minnesota, Minneapolis, Minn.
WARD, Henry L. (June 30, '16) Chem. Eng., Western Elec. Co., Chicago, Ill.; mailing address, 146 S. Hamlin Ave.
WARD, Louis E. (Mar. 25, '08) Box 94, Midland, Mich.

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WATERMAN, Frank N. (Apr. 3, '02) 100 Broadway, New York City.
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WATTS, Oliver P., Ph.D. (Mar. 5, '04) Asst. Prof. of Applied Electrochem., Univ. of Wisconsin; res., 114 Sponner St., Madison, Wis.
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WEBER, Dr. M. G. (Apr. 29, '11) 76 Rutland Road, Brooklyn, N. Y.
WEBERT, Louis P. (Feb. 23, '12) Chem., Engincering Experiment Station, Annapolis, Md.; mailing address, 139 Charles St.
WEBSTER, Clement L. (Apr. 24, '09) Geologist and Mining Engr., The Interstate Investment and Development Co., Charles City, Floyd Co., Iowa; mailing address, 111 Hawkins Ave.
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WILLIAMS, Frank M. (June 28, '12) Consulting Chemical Engr., 177 Mullin St., Watertown, N. Y.; res., 43-44 Sherman Bldg., Watertown, N. Y.
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WINTER, E. J. (Apr. 22, '15) U. S. Industrial Alcohol Co., Curtis Bay, Md.
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WISE, John B. (Feb. 24, '17) Supt. Electrolytic and Sulphuric Plants, Chile Exploration Co., Chuquicamata, Chile.
WITHERELL, Chas. S. (July 25, '13) Met. Eng., care of The Chile Exploration Co., 120 Broadway, New York City.
WITHERSPOON, R. A. (Oct. 2, '02) Shawinigan Carbide Co., Shawinigan Falls, Quebec, Canada.
WITHERSPOON, T. A. (Apr. 3, '02) Nat. Union Bldg., 918 F St., Washington, D. C.
WITHROW, James R. (May 29, '09) Prof. Industrial Chemistry, Ohio State University, Columbus, Ohio.
WOHLWILL, Dr. Heinrich (June 29, '07) Norddeutsche Raffinerie, Elbstr. 35, Hamburg, Germany.
WOLF, Harry J. (Sept. 7, '06) Min. Eng., First National Bank Bldg., Denver, Colo.; mailing address, 1510 Washington Ave., Golden, Colo.

WOOD, E. F. (Apr. 3, '02) Vice-Pres., Int. Nickel Co., 43 Exchange Pl., New York City.
WOOD, Prof. H. P. (Jan. 5, '07) Prof. Elec. Eng., Georgia Technical School, Atlanta, Ga.
WOODRUFF, John C. (June 30, '17) Res. Chem., Western Elec. Co., 463 West St., New York City.
WOODSIDE, F. C. (Apr. 26, '13) Production Eng., General Elec. Co., Pittsfield, Mass.; res., 831 North St.
WOODWARD, J. M. (May 6, '05) Nela Park, Cleveland, Ohio.
WOODWORTH, S. E. (Oct. 22, '15) Metallurgist, with Chas. Butters, 419 Embarcadeso, San Francisco, Cal.
WOOLDRIDGE, Wm. J. (Nov. 21, '08) Gen. Elec. Co., 134 Appleton Ave., Pittsfield, Mass.
WORTH, B. G. (May 26, '10) Electrical Engineer, Walter Kidde, Engineer-Contractor, 120 Cedar St., New York City; residence, 954 Kensington Ave., Plainfield, N. J.
WRIGHT, Arthur (Apr. 27, '12) Lawyer, Firm of Prindle, Wright & Small, 111 Broadway, New York City; res., 45 Markwood Road, Forest Hills Garden, L. I., N. Y.
WRIGHT, J. C. (Sept. 15, '15) 48 West 94th St., New York City.
WRINKLE, Noah (Sept. 27, '16) Consult. Eng., 2599 Sacramento St., San Francisco, Cal.
WURSTER, Oscar H. (May 27, '11) Supt., Louisville Soap Co., Louisville, Ky.
WYMAN, Walter S. (Mar. 27, '14) Treasurer and Gen. Mgr., Central Maine Power Co., 49 Western Ave., Augusta, Maine.
YAGER, John J. (Dec. 31, '15) Sec'y of Goergen-Mackwirth Co., Inc.; mailing address, 268 Carlton St., Buffalo, N. Y.
YAMASAKI, Dr. Jingoro (Apr. 29, '11) The Imperial Industrial Lab., Yetchiushima, Tokyo, Japan.
YARDLEY, John L. (Aug. 25, '17) Gen. Eng., Westinghouse Elec. & Mfg. Co.; mailing address, 1263 Denniston Ave., Pittsburgh, Pa.
YATSEVITCH, Michael (Feb. 23, '12) Chief Eng., Government Committee on Fuels, Vasil Ost. 2, 11 Line N. 52, 16, Petrograd, Russia.
YNGSTROM, Lars (Dec. 31, '10) Owner of Sandsta Elektriska Smältwerk, Haggo, Sweden.
YOSHIKAWA, Prof. Kamejiro (Dec. 27, '07) Nakasuji Ishuyakushi, Kyoto, Japan.
YOUNG, Hayes W. (July 25, '13) Asst. Prof. of Met., Stanford Univ., Stanford University P. O., Cal.
YOUNG, John (Oct. 26, '17) Mgr. and Partner, Box 858, Welland, Ontario, Canada.
YOUNT, Andrew S. (June 1, 1915) Supervisor Explosive Mfg. Operating Dept., Du Pont Powder Co., Deepwater Point, N. J.; mailing address, 1022 Jackson St.
YUNCK, Carl I. (Oct. 21, '16) Supt., Republic Elec. Co.; mailing address, 422 Meeker St., South Orange, N. J.
YUNCK, John A. (Apr. 30, '08) Electrometallurgist, 375 Ogden St., Newark, N. J.; mailing address, 422 Meeker St., South Orange, N. J.
ZAREMBA, Edward (Jan. 28, '08) 707 D. S. Morgan Bldg., Buffalo, N. Y.
ZELLER, Richard (Apr. 24, '14) Factory Mgr., Egyptian Lacquer Mfg. Co., Rahway, New Jersey; mailing address, 595 West End Ave., New York City.
ZIMMERMAN, J G. (Nov. 6, '02) Service Eng., 1466 S. Michigan Ave., Chicago, Ill.
ZIMMERMAN, R. E. (Jan. 26, '17) Asst. Director, Res. Lab., American Sheet & Tin Plate Co.; mailing address, 210 Semple St., Pittsburgh, Pa.
ZIMMERMANN, Dr. Fritz (June 29, '07) care of Baker & Co., Inc., Platinum Works, Murray St., Newark, N. J.
ZWINGENBERGER, Otto K. (Jan. 27, '12) Chemist, Patent Dept., Roessler & Hasslacher Chem. Co., Perth Amboy, N. J.

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Parr, S. W.

Winthrop Harbor—
Aiken, R. H.

INDIANA

Bloomington—
Brown, O. W.
Mathers, F. C.

East Chicago—
Bonsib, R. S.
Mulligan, J. J.

Hammond—
Thum, Wm.

Lafayette—
Duncan, T.

Terre Haute—
Booth, W. K.

West Lafayette—
Unger, G. N.

IOWA

Ames—
Mann, C. A.

Boone—
Mottinger, B. T.

Charles City—
Webster, C. L.

Iowa City—
Pearce, J. N.

Keokuk—
Baker, R. E.
Davis, H. N.
Knight, F. P. H.
Roll-Hansen, C.
Scholl, G. D.
Weissenburger, G. E.
Wettstein, T. F.

KENTUCKY

Louisville—
Ruby, C. E.
Worster, O. H.

Marion—
Reed, A. H.

Newport—
Patrick, R. C.

LOUISIANA

Destrehan—
Gould, D. F.

New Orleans—
Williamson, C. S., Jr.

MAINE

Augusta—
Wyman, W. S.

Cumberland Mills—
Gabriel, G. A.

Great Works—
Larchar, A. B.

Lewiston—
Robinson, A.

Portland—
Allen, H. I.
Lincoln, E. S.
McMurtrie, D. H.
Moulton, S. A.

Rumford—
Griffin, Martin L.

South Brewer—
Hanson, H. H.

MARYLAND

Annapolis—
McAdam, D. J., Jr.
Webert, L. P.

Baltimore—
Bate, H. A.
Boynton, W. H.
Bryan, J. K.
Falter, P. H.
Lovelace, B. F.
Peirce, W. H.
Rouse, E. W., Jr.
Tillman, R. H.
Weitlaner, R. J.

Camp Meade—
Starr, A. B., Jr.

Chevy Chase—
Schramm, Edw.

Colgate—
Aldrich, C. H.

Cumberland—
McKaig, W. W.

Curtis Bay—
Winter, E. J.

Emmitsburg—
Rauth, J. W.

Luke—
Pedder, John

MASSACHUSETTS

Boston—
Barker, E. R.
Buchanan, L. B.
Clapp, E. H.
Eustis, A. H.
Kao, T.
Kniffin, L. M.
Pratt, F. S.
Sargent, F. C.
Sharples, S. P.
Spaulding, H. W.
Stone, C. A.
Webster, E. S.
Wentworth, H. A.
Werby, A. B.

MASSACHUSETTS—Continued

Brookline—
Howard, H.
Walker, E. C., 3d

Cambridge—
Comstock, D. F.
Forbes, Geo. S.
Goodwin, H. M.
Hurum, Fredrik
Kalmus, H. T.
Lamb, A. B.
Little, A. D.
MacInnes, D. A.
Nickerson, W. E.
Richards, T. W.
Skinner, H. J.
Talbot, H. P.
Thompson, M. deK.
Walker, Wm. H.

Gloucester—
Dorsey, H. G.
Hills, L. H.

Lowell—
Shepherd, F. A.

Lynn—
Coates, Jesse
Miller, L. B.
Nestor, J. F.

Malden—
Berry, E. R.

Marlboro—
McManus, Jos. D.

Norwood—
Treacy, C. S.

Pittsfield—
Frank, J. J.
Gifford, A. McK.
Kelly, J. F.
Unger, M.
Woodside, F. C.
Wooldridge, W. J.

Southbridge—
Reeve, H. T.

Springfield—
Morris, A. W.

Swampscott—
Fleming, R.
Thomson, E.

Taunton—
Howe, David

Waltham—
Flagg, F. P.

Williamstown—
Mears, Brainerd

Winchester—
Marsh, C. W.

Wollaston—
Ruppel, H. E. K.

Worcester—
Calhane, D. F.
Higgins, A. C.
Jeppson, G. N.

MICHIGAN

Ann Arbor—
Badger, W. L.
Bartell, F. E.
Bigelow, S. L.
Willard, H. H.

Bay City—
Hutchins, C. F.

MICHIGAN—Continued

Detroit—
Caulkins, E. B.
Cole, C. S.
Cowperthwait, A. D.
Crosby, E. L.
Harig, Fred. C.
Hirshfeld, C. F.
Jack, Geo. B.
Lane, H. M.
Lohr, J. M.
Marsh, A. L.
Ramage, A. S.
Schmelz, E. M.
Skillman, Verne
Stephenson, F. T. F.
Stewart, R. S.
Stotter, Max

Flint—
Copeman, L. G.

Ironwood—
Lathrop, L. H.

Menominee—
Sims, C. E.
White, A. R.

Midland—
Burdick, E. C.
Dow, H. H.
Ward, L. E.

White Pigeon—
Unzicker, S. P.

Wyandotte—
Cranston, John
Hardcastle, Y. F.

MINNESOTA

Duluth—
Oldfield, Lee W.

Minneapolis—
Frankforter, G. B.
Hunter, J. V.
Miller, L. F.
Somdal, J. A.
Ward, H. L.

St. Paul—
Temple, Sterling

MISSOURI

Columbia—
Brown, W. G.
Schlundt, Herman

Joplin—
Wallower, F. C.

Kansas City—
Cherry, L. B.
Kent, J. M.

St. Louis—
Curfman, F. G.
Kohler, H. L.
Queeny, J. F.
Ruffner, C. S.

MONTANA

Anaconda—
Frick, F. F.

Billings—
Heath, R. F.

Butte—
Bowman, C. H.
Deshler, G. O.
Pulsifer, H. B.

MONTANA—Continued

Great Falls—
Burns, W. T.
Krejci, Milo W.

Helena—
Gerry, M. H., Jr.

NEBRASKA

Omaha—
Hall, A. E.
Phillips, Ross
Randall, A. G.
Reinbold, Herman

NEVADA

Lovelock—
Reid, John T.

Searchlight—
Barton, W. H.

Virginia City—
Symmes, W.

NEW HAMPSHIRE

Berlin—
Barton, C. B.
Comstock, R. L.
Fogarty, J. A.
Moore, H. K.
Richter, G. A.

Durham—
Perley, G. A.

Wilton—
Abbott, Wm. G., Jr.

NEW JERSEY

Aldene—
Hayes, G. W.

Arlington—
Pickering, O. W.
Proctor, C. H.

Bayonne—
Burgess, Louis
Ramsey, F. H.
Schuyler, A. H.

Bloomfield—
Amer, H. S.
MacRae, Duncan
Romanelli, E.
Vassar, H. S.

Bound Brook—
Hemingway, F.

Camp Dix—
Whitlock, Maj. E. H.

Carteret—
Breckenridge, J. E.

Chrome—
Deacon, R. W.
Green, H. M.
Greenwood, H. D.
Hood, B. B.
Smith, W. C.

Dover—
Akinfieff, Boris

East Orange—
Arthur, Paul
Cowles, Harry D.
Doubleday, R. S.
Koerner, W. E.
Needham, H. H.

NEW JERSEY—Continued

Elizabeth—
Hendrie, G. A.
Martens, Paul
Patterson, T. A.
Pyne, F. R.

Essex Falls—
Robb, Chas. D.

Gloucester City—
Knoedler, E. L.
Miner, H. S.

Hackensack—
Schulze, Paul

Harrison—
Hart, L. O.

Haskell—
Cummings, Wm. J.

High Bridge—
Hall, John H.
Le Boutillier, Clement

Hoboken—
Bijur, Jos.
Winninghoff, W. J.

Irvington—
Bachofner, D. K.

Jamesburg—
Foersterling, Hans

Jersey City—
Baldwin, A. I.
Hartwick, Frank A.
Lubowsky, Simon J.
Sieger, G. N.
Swartley, H. R., Jr.

Maurer—
Alexander, H. H.
Mindeleff, Chas.

Merchantville—
Wickes, C. S.

Montclair—
Crane, F. D.
Ellis, Carleton
Smith, Dyer

Newark—
Benjamin, E. O.
Boice, E. N.
Burger, Alfred
Campbell, P. A.
Carter, F. E.
Colby, E. A.
Dewey, E. S.
Driver, W. B.
Drobegg, G.
Fernberger, H. M.
Foster, R. D.
Gifford, W. E.
Heath, H. E.
Henderson, C. T.
Hoffmann, John
Hubley, W. F.
Kleinfeldt, H. F.
Liebschutz, M.
Madsen, C. P.
McNeill, Ralph
Metz, G. P.
Pope, R. W.
Robinson, F. W.
Sievering, Philip
Walsh, P. C., Jr.
Weston, E.
Williams, N. O.
Zimmermann, F.

NEW JERSEY—Continued

New Brunswick—
Keller, Oran
Voorhees, L. A.

North Bergen—
Van Keuren, W. L.

Orange—
Edison, T. A.
Kammerhoff, H. H.
Kennedy, A. M.
Smith, H. H.

Paterson—
Jarvis, E. G.

Perth Amboy—
Antisell, F. L.
Brown, M. J.
Fisher, H. W.
Kaufmann, F. A.
McNitt, R. J.
Philipps, H.
Roessler, F.
Rossi, Louis M.
Skowronski, S.
Stewart, M. E.
Waring, T. D.
Zwingenberger, O. K.

Phillipsburg—
Baker, John T.

Plainfield—
Hibbard, H. D.
Manahan, Paul R.
Pratt, H. A.
Spicer, C. W.
Worth, B. G., Jr.

Princeton—
Hulett, G. A.
Northrup, E. F.
Taylor, H. S.

Rahway—
Breckenridge, C. E.
Maeulen, Frederick
Miller, Daniel
Murray, B. L.

Roselle—
Grymes, E. S.

Rutherford—
Daft, Leo
Schroeder, C. M. E.

Sewaren—
Buttfield, W. J.
Cowles, A. H.
Lemberg, M.

South Orange—
Yunck, Carl I.
Yunck, J. A.

Summit—
Hornsey, J. W.

Trenton—
Redfield, C. S.
Schleeder, L. B.

Upper Ridgewood—
Czarnecki, F. C.

Watchung—
Moldenke, Richard

Weehawken—
Kraus, Ernest
Laise, C. A.
Schubert, B. H.

West Orange—
Pedersen, A. Z.

Woodbridge—
Child, H. A.

NEW YORK

Albany—
Rankin, Herbert E.

Astoria (L. I.)—
Mahlman, O. L.
Wilson, I. W.

Auburn—
Case, T. W.
Case, W. E.

Baldwin (L. I.)—
Vogeler, W. R.

Bedford Hills—
Howe, H. M.

Beechurst (L. I.)—
Landis, W. S.

Brooklyn—
Allen, O. F.
Carrier, S. C.
Cook, R. J.
Cowan, Wm. A.
Erhart, W. H.
Foster, O. R.
Frederick, G. E., Jr.
Kemper, D. A.
Nees, A. R.
Reich, J. S.
Scott, L. C.
Sheldon, S.
Sperry, E. A.
Voss, William
Watkins, W. H.
Weber, Max G.

Buffalo—
Albright, L.
Bierbaum, C. H.
Carrier, W. H.
Corse, Wm. M.
Curtiss, John L.
Cushing, H. M.
Doty, E. L.
Fish, Job, Jr.
Hardie, Chas. G.
Jones, A. C.
Lawrence, J. N.
Mattern, G. G.
Neal, J. R. H.
Patch, N. K. B.
Rippel, E. G.
Schoellkopf, J. F., Jr.
Schranz, T. L.
Sicard, H. C.
Stephenson, H. L.
Strachan, E. K.
Theurer, G. A.
Underwood, C. W.
White, H. E.
Wilke, Wm.
Wilson, J. R.
Yager, J. J.
Zaremba, Edward

Clinton—
Saunders, A. P.

Flushing (L. I.)—
Bajda, J. J.
Lyons, H. N.

Fort Plain—
Fox, W. J.

Fulton—
Valentine, I. R.

Glens Falls—
Hilliard, John D.

NEW YORK—Continued

Ithaca—
Bancroft, W. D.
Gillett, H. W.
Mack, E. L.
McBerty, F. H.
Thompson, J. G.
Williams, C. E.

Kenwood—
Reeve, A. G.

LaSalle—
Shields, J. E.

Lockport—
Howard, L. E.
Kenan, W. R., Jr.

Massena—
Doerschuk, V. C.
Hall, H. M.
Little, W. T.

Menands—
Shiverick, M. D.

Mt. Vernon—
Brown, C. J.
Wilson, C. H.

New York City—
Abbé, P. O.
Acheson, E. G.
Addicks, L.
Allyn, R. S.
Aldridge, W. H.
Andrews, Mary R.
Anger, E. M.
Atwater, C. G.
Baker, H. A.
Barnes, H. H., Jr.
Barrows, F. E.
Barstow, W. S.
Baskerville, C.
Beck, E. A.
Bedell, E. H.
Bergen, R. C.
Boeck, P. A.
Bogue, C. J.
Bowman, W.
Bradley, Linn
Bradley, W. E. F.
Breneman, A. A.
Brown, H. P.
Buck, H. W.
Cameron, W. S.
Canet, B. Chas.
Carse, D. B.
Castle, S. N.
Chandler, C. F.
Clark, W. G.
Clark, W. J.
Coho, H. B.
Cohoe, W. P.
Cooper, K. F.
Corning, C. R.
Crocker, J. R.
De Miles, Paul
Deppe, W. P.
Dixon, Jos. L.
Doerflinger, W. F.
Doremus, C. A.
Dorr, J. V. N.
Douglas, James
Dreyfus, W.
Dyrssen, Waldemar
Eagle, H. Y.
Eimer, A.
Emerson, H.
Englehard, C.
Eurich, E. F.

NEW YORK—Continued

New York City—Continued

Fink, C. G.
FitzGibbon, R.
Flashman, H. W.
Fliess, R. A.
Foley, C. B.
Frank, K. G.
Freas, Thos. B.
Fries, H. H.
Gaines, R. H.
Glenn, E. R.
Goepel, C. P.
Gray, J. H.
Grosvenor, W. M.
Guess, H. A.
Guiterman, K. S.
Halcomb, C. H.
Harris, J. W.
Hasslacher, J.
Hatzel, J. C.
Hendry, W. F.
Herty, C. H.
Hill, N. S., Jr.
Hirsch, Alcan
Hirschland, F. H.
Hoeft, G. Eliot
Hoge, J. F. D.
Hunt, A. M.
Huntoon, L. D.
Ingalls, W. R.
Inui, Kyujiro
Jacoby, H. E.
Jinguji, Genjiro
Johnson, J. E., Jr.
Keller, Edw.
Kennedy, J. J.
Kern, E. F.
Kingsley, E. D.
Klipstein, E. C.
Knapp, Geo. O.
Kohn, M. M.
Kunz, G. F.
Landau, Alfred
Langton, J.
Leavitt, Wm. F. B.
Ledoux, A. R.
Leslie, E. H.
Liebmann, A. J.
Lindsay, C. F.
Loebell, H. O.
Love, E. G.
Lovejoy, D. R.
MacDonald, J. A.
Magnus, Benjamin
Maier, C. G.
Mailloux, C. O.
Mantius, Otto
Martin, T. C.
Marvin, A. B.
Mason, F. S.
Mastick, S. C.
Mays, S. W.
Maywald, F. J.
McMahon, G. F.
Mershon, R. D.
Metz, H. A.
Miller, D. D.
Mohn, Arnold
Moody, H. R.
Morey, S. R.
Morgan, J. L. R.
Morrow, J. T.
Morse, W. S.
Mortimer, J. D.
Muir, J. M.
Muschenheim, F. A.

NEW YORK—Continued

New York City—Continued

Myers, W. S.
Nagelvoort, Adriaan
Nichols, W. H.
Nichols, W. S.
Ornstein, G.
Osborne, L. A.
Oshima, Yoshikiyo
Parmelee, H. C.
Paulsson, Axel
Pennie, J. C.
Peters, F. F.
Petinot, N.
Price, E. F.
Prindle, E. J.
Prosser, H. A.
Raimondo, Sebastiano
Reber, Samuel
Reed, S. A.
Rhodin, P. E. F.
Ricketts, L. D.
Riglander, M. M.
Riker, J. J.
Riley, L. A., 2d
Roberts, G. J.
Roller, F. W.
Roth, C. F.
Rowand, L. G.
Ruhl, L.
Schildhauer, Edw.
Schloss, J. A.
Schuetz, F. F.
Seward, G. O.
Sharp, C. H.
Smith, E. A. Cappelen
Speed, Buckner
Speiden, C. C.
Spilsbury, E. G.
Statham, Noel
Stevenson, Reston
Stone, I. F.
Stone, Jos. P.
Stone, J. S.
Stoughton, Bradley
Swenarton, W. H.
Takamine, J.
Teeple, J. E.
Thatcher, C. J.
Thompson, J.
Toch, M.
Treadwell, J. C.
Tucker, S. A.
Van Arsdale, G. D.
Vom Baur, C. H.
Von Foregger, Richard
Waddell, M.
Walker, A. L.
Washburn, F. S.
Waterman, F. N.
Weeks, F. D.
Weintraub, Ezechiel
Wells, J. S. C.
Westervelt, W. Y.
Wheeler, A. E.
Whitaker, M. C.
White, J. G.
White, J. H.
Wiechmann, F. G.
Wigglesworth, H.
Wilkinson, W. N.
Williams, A.
Williams, J. T.
Williams, L. W.
Winship, W. E.
Witherell, C. S.
Wood, E. F.

NEW YORK—Continued

New York City—Continued

Woodruff, J. C.
Wright, Arthur
Wright, J. C.
Zeller, R.

Niagara Falls—

Baker, E. M.
Barton, P. P.
Bayard, R. A.
Beard, C. W.
Becket, F. M.
Blanchard, H. J.
Bliss, Wm. Lord
Carveth, H. R.
Chace, R. T.
Cole, E. R.
Converse, V. G.
Cox, G. E.
Dunlap, O. E.
Edmands, I. R.
FitzGerald, F. A. J.
Fowler, R. E.
Frary, F. C.
Fuller, G. P.
Gegenheimer, R. E.
Geyler, F. O.
Gilbert, H. N.
Glaze, John B.
Glennie, R. D.
Griffith, J. R.
Hamann, A. M.
Harper, J. L.
Herzog, G. K.
Hibbert, Harold
Hinckley, A. T.
Hooker, A. H.
Hutchins, Otis
Imlay, L. E.
Jacobson, B. H.
Johnson, J. A.
Kellogg, H. W.
Koethen, F. L.
Kokatnur, V. R.
Lansing, C. N.
Lavene, H. A.
Lebherz, John
Leffel, C. E.
Lidbury, F. A.
Low, F. S.
Lowe, R. E.
Lyster, T. L. B.
MacMahon, J. D.
MacMahon, J.
MacMillan, J. R.
Marshall, J. G.
Mauran, M.
McKnight, W. A.
Meredith, W. F.
Moritz, C. H.
Moyer, G. C.
Mueser, Emil
Neuhaus, R.
Noyes, H. L.
Nutting, E. G.
Osborne, S. E.
Ralston, O. C.
Rossi, A. J.
Rowland, J. M.
Saunders, L. E.
Schabacker, H. E.
Schoellkopf, P. A.
Sergeant, E. M.
Simmers, A. L.
Smith, A.
Smith, E. S.
Snowdon, R. C.

NEW YORK—Continued

Niagara Falls—Continued

Sneiden, E. C.
Stamps, F. A.
Stillesen, J. M. A.
Stone, G. W.
Tone, F. J.
Vaughn, C. F.
Wallace, W.
Wescott, E. W.
Whitaker, A. W., Jr.
White, R. H.
Williamson, A. M.
Wilson, N. A.
Winder, C. A.

Oneida—

Bailey, R. O.

Ossining—

Acker, Chas. E.

Piermont—

Main, W.

Penn Yan—

Taylor, C. E.

Plattsburg—

Sheffield, W. T.

Prince Bay (L. I.)—

Johnston, F. A.
Johnston, W. A.

Richmond Hill (L. I.)—

Haslwanter, C.
Herreshoff, J. B.

Rochester—

Lee, I. E.
Schwarz, R. C.
Will, R. T.

Rome—

Parish, R. R.

Schenectady—

Andrews, W. S.
Arsem, W. C.
Berg, E. J.
Coffin, F. P.
Coolidge, Wm. D.
Creighton, E. E.
Dantsizen, Christian
Dushman, Saul
Hawkins, L. A.
Langmuir, I.
Lof, E. A.
Murphy, E. J.
Reist, H. G.
Rushmore, D. B.
Seede, J. A.
Steinmetz, C. P.
Taylor, J. B.
Thompson, M. R.
Thurston, L. S.
Van Brunt, C.
Whitney, W. R.
Willey, L. M.

Syracuse—

Brookfield, W. B.
Conklin, E. B.
Cooper, H. C.
Harvey, F. A.
Matthews, J. A.
Newkirk, E. D.
Pennock, J. D.

Troy—

Bryson, T. A.
Hunter, M. A.
Lincoln, A. T.
Patton, D. C.

NEW YORK—Continued

Webster—
Weeks, F. D.

Yonkers—
Baekeland, L. H.
Duurloo, F.

NORTH CAROLINA

Asheville—
Betts, A. G.

Charlotte—
Gilchrist, P. S.

Clemmons—
Pickens, Rufus H.

West Raleigh—
Browne, W. H.

Whitney—
Kemmer, F. R.

OHIO

Akron—
Shaw, E. C.

Alliance—
Baily, T. F.
Cope, F. T.
Foster, C. L.

Amherst—
Briggs, F. H.

Barberton—
Austin, A. O.

Canton—
MacGregor, Walter
Meyer, R.

Cincinnati—
Davison, A. W.
Dittmar, Carl
Ecker, Howard, Jr.
Elliott, Geo. K.
Roessler, E. B.

Cleveland—
Brown, J. W.
Burwell, A. W.
Chillas, R. B., Jr.
Clymer, W. R.
Cook, Edw. B.
Crider, J. S.
Drefahl, L. C.
Fleming, S. H.
Graves, W. G.
Holmes, M. E.
Hyde, E. P.
Koehler, W.
Kranz, W. G.
Mainwaring, Wm. D.
Megroot, J. P.
Merrill, G. S.
Morrison, G. O.
Orr, C. A.
Pratt, E. B.
Richardson, E. A.
Schmidt, J. H.
Smith, A. W.
Tower, O. F.
Wellman, S. T.
Woodward, J. M.

Columbus—
Daines, W. P.
Demorest, D. J.
Leonard, G. M.
Schaaf, Downs
Withrow, J. R.

OHIO—Continued

Dayton—
Clements, F. O.
Gestle, John
Niswonger, E. E.
Suman, R. G.

Fostoria—
Downes, A. C.

Fremont—
Goodwin, J. H.

Lakewood—
Brooks, W. C.
Bullock, A. R.
Chaney, N. K.
Chapin, H. C.
Gillingham, C. A.
Hazelett, C. W.
Mann, W. W.
Moore, Wm. C.
Mott, W. R.
Ordway, D. L.
Pulman, O. S.
Reid, R. R.
White, F. L.

Lima—
Arthur, Walter

Mansfield—
Darrah, W. A.

Massillon—
Kennedy, T. O.

Middletown—
Ahlbrandt, G. F.
Eldridge, S. E.

Napoleon—
Long, G. E.

Oberlin—
Crafts, W. N.

Portsmouth—
Kinnear, H. B.

Rittman—
Laib, Walter

Salem—
Davis, D. L.

Toledo—
Fawcett, L. H.
Green, S. S.
Nagel, W. G.
Wenner, R. S.

Wadsworth—
Shapiro, H.

OKLAHOMA

Bartlesville—
Born, Sidney
Parkhurst, I. P.

Shawnee—
Dodge, W. E.

Tulsa—
Miller, Walter
Taber, G. H., Jr.

OREGON

Cornucopia—
Nestler, G. A.

Hood River—
Butcher, J. C.

Marshfield—
Mereen, J. D.

Portland—
Hall, E. L.

PENNSYLVANIA

Allentown—
McCullough, H. F.

Altoona—
Casselberry, H.

Ambridge—
Farnham, F. F.
Meineke, O. H.

Ardmore—
Brumbaugh, A. K.

Aspinwall—
Hedden, S. E.
Hessom, B. F., Jr.
McMillen, R. H.

Bellefonte—
Ward, A. T.

Ben Avon—
Aston, J.

Bethlehem—
Blasius, C. E.
Buck, C. A.
Butts, Allison
Drinker, P. H.
Fehnel, J. Wm.
Flinn, E. H.
Hommel, R. P.
MacNutt, Barry
Mitman, W. T.
Richards, J. W.
Roush, G. A.
Seyfert, S. S.
Takikawa, Yasuo

Boyerstown—
Corbin, J. R.

Brackenridge—
Connell, H. R.
Ober, J. E.

Braddock—
Lewis, J. D.

Bridgeville—
Saklatwalla, B. D.

Canonsburg—
Vogt, L. F.

Carnegie—
Ohlwiler, C. H.
Reese, P. P.

Chester—
Comey, A. M.
Krause, W. B.

Conshohocken—
Davis, F. W.

Coraopolis—
Hensen, Emil
Stafford, S. G.

Crafton—
Griffin, G. B.

Creighton—
Gelstharp, F.

Duquesne—
Stewart, H. M.

Easton—
Adamson, G. P.
Buhl, Wm.
Gordon, C. McC.
Hart, E.
Shimer, E. B.

Edgewood Park—
Scott, W. S.

PENNSYLVANIA—Continued

East Pittsburgh—
Schoepf, T. H.
Skinner, C. E.

Ellwood City—
Dunn, J. J.
Offutt, J. W.

Gettysburg—
Parsons, L. A.

Greensburg—
Koppitz, C. G.

Harrisburg—
Calder, A. R.
Kinter, G. R.

Houston—
Patterson, C. T.

Jenkintown—
Davis, R. W., Jr.

Johnsonburg—
Buckie, H. H.

Johnstown—
Parkhurst, C. W.

Latrobe—
Garratt, Frank
Knox, L. B.

Lebanon—
Weimer, E. A.

McKeesport—
Goodspeed, G. M.

Monessen—
Owens, E. W.

Mount Penn—
Kramer, L. B.

Munhall—
Hopkins, G. A.

Natrona—
Darlington, H. T.

New Castle—
Lamker, H. G.
Samuels, Wm. P.

New Kensington—
Hamor, W. A.

Newton Hamilton—
Snyder, J. L. K.

Oakmont—
Fernekes, Gustave
Hitner, H. F.
Moore, J. R.

Palmerton—
Holstein, L. S.
Smull, J. G.

Parnassus—
Blough, Earl

Philadelphia—
Asef, Waldemar
Benoliel, S. D.
Bonine, Chas. E.
Breed, G.
Brophy, O.
Brown, R. P.
Buch, N. W.
Burman, B. F.
Bustos, Enrique
Callen, A. S.
Cary, C. R.
Clamer, G. H.
Devereux, W.
Eglin, W. C. L.
Fraley, J. C.

PENNSYLVANIA—Continued

Philadelphia—Continued

Franklin, M. W.
Furness, R.
Gerety, J. J.
Gibbs, A. E.
Hall, C. A.
Hering, C.
Hess, H.
Hicks, Edwin F.
Hildebrand, J. H.
Hitchcock, F. R. M.
Holland, W. E.
Howard, G. M.
James, W. F.
Keith, N. S.
Kitsee, I.
Kutz, Milton
Lafore, J. A.
Lavino, E. J.
Lay, J. Tracy
Lukens, H. S.
McConnell, J. Y.
Merzbacher, Aaron
Meyer, J.
Moerk, F. N.
Morgan, Leonard P.
Ogden, John
Paul, H. N.
Rittenhouse, E.
Rosengarten, G. D.
Russell, C. J.
Sadtler, S. P.
Sadtler, S. S.
Salom, P. G.
Schamberg, M.
Smith, E. F.
Smith, E. W.
Smith, H. F., Jr.
Stevens, J. F.
Taggart, W. T.
Taylor, H. N.
Thwing, C. B.
Weeks, C. A.
Weisenburg, Andrew
Wells, H. P.
Williams, H. J.
Wilson, T. E.
Wirt, Chas.

Pittsburgh—

Bacon, R. F.
Bear, S. L.
Brown, John T., Jr.
Clarke, E. B.
Crabtree, F.
Crawford, C. A.
Darrin, Marc
Dewey, B.
Dougherty, J. W.
Flannery, Jas. J.
Gibson, C. B.
Goodale, S. L.
Hartley, R. H.
Hitchcock, H. K.
James, J. H.
Jones, G. W.
Kemery, P.
Kier, S. M.
Laughlin, H. H.
Lincoln, P. M.
Loeffler, Geo. O.
Mathias, D. L.
McDonald, R. A.
McKinley, Jos.
McNiff, G. P.
Meyers, H. H.
Moore, W. E.

PENNSYLVANIA—Continued

Pittsburgh—Continued

O'Neil, R. D.
Page, G. S.
Palmer, C. S.
Pinkerton, A.
Pluck, A. F.
Pope, Chas. E.
Ray, H. C.
Rodman, H.
Schluederberg, C. G.
Slocum, F. L.
Snyder, C. G.
Speller, F. N.
Stahl, N.
Stewart, R. T.
Stone, E. C.
Stupakoff, S. H.
Styri, Haakon
Tafel, Theodore, Jr.
Tiemann, H. P.
Turnock, E. H., Jr.
Turnock, L. C.
Uhlenhaut, F., Jr.
Unger, J. S.
Viol, C. H.
Wadsworth, F. L. O.
Weidlein, E. R.
Wiley, B.
Yardley, J. L. McK.
Zimmerman, R. E.

Primos—

Boericke, G.
Stein, W. M.

Reading—

Parker, Jas. H.
Shindell, H. F.

Roaring Springs—

McDonald, F.

Slatington—

Eberwein, S. J.

Springdale—

Cox, S. F.

State College—

Bright, A. C.
Chedsey, W. R.
Dudley, Boyd, Jr.
Pond, G. G.

Steelton—

Reed, J. C.

Swarthmore—

Alleman, G.
Creighton, H. J.

Tamaqua—

Burt, M. C.
Given, G. C.
McQuaid, H. S.

Tarentum—

Parkinson, J. C.

Washington—

Duval, A. L.
Morrison, M. E.
Sargent, G. W.

Wilkinsburg—

Adams, J. L., Jr.
Bjorkstedt, G. W.
Stephens, C. E.
Wills, Wm. H., Jr.

PENNSYLVANIA—Continued

Wilkes-Barre—
Strauss, F. A.

Wyomissing—
Parker, J. H.

York—
Devers, P. K.

RHODE ISLAND

Newport—
Richards, E.

Providence—
Andrews, J. H.
Saunders, W. M.
Thurber, J. H.

SOUTH CAROLINA

Columbia—
Mills, J. E.

Sumter—
Van Deventer, H. R.

TENNESSEE

Allens Creek—
Foust, T. B.

Maryville—
Adams, T. J.
Parks, R. E.

Mascot—
Toomer, J. E.

TEXAS

Austin—
Harper, H. W.
Schoch, E. P.

Fort Worth—
Slater, W. A.

Houston—
Weiser, H. B.

Port Arthur—
Alexander, C. M.
Stockton-Abbott, L.

UTAH

La Sal—
Morgan, H. J.

Pleasant Grove—
Hayes, J. J.

Provo City—
Smith, F. W.

Salt Lake City—
Austin, L. S.
Blossom, E. L.
Bradford, R. H.
Emery, W. L.
Hansen, C. A.
Koering, B. K.
Putnam, W. R.

VERMONT

Northfield—
Howard, S. F.

VIRGINIA

Charlottesville—
Weaver, W. D.

Holcomb's Rock—
Lee, H. R.

VIRGINIA—Continued

Norfolk—
Webb, L. W.

Portsmouth—
Barclay, E. H.

Richmond—
Walters, Jos.

University—
Dunnington, F. P.

Yorktown—
Williams, E. R.

WASHINGTON

Seattle—
Miller, A. A.
Ryan, F. C.
Thomas, B.

Spokane—
Armstrong, L. K.
Keffer, Frederick

Tacoma—
Everette, W. E.
Morse, W. V.
Porro, T. J.

Valley—
Bowman, F. C.

WEST VIRGINIA

Charleston—
Carrier, C. F., Jr.
Pierce, J. B., Jr.
Rollin, Hugh

Follansbee—
Barr, H. L.

Morgantown—
Clark, F. E.
Hite, B. H.

Piedmont—
Randall, J. W. H.

St. Albans—
Sargent, R. N.

Warwood—
Bumgardner, J. W.

WISCONSIN

Madison—
Burgess, C. F.
Hamister, V. C. H.
Helfrecht, A. J.
Kahlenberg, L.
Kowalke, O. L.
Ruhoff, O. E.
Schulte, W. B.
Storey, O. W.
Watts, O. P.

Milwaukee—
Bovee, B. A.
Gosrow, R. C.
Kremers, J. G.
Raeth, F. C.
Richardson, L. T.
Smith, J. H.

WYOMING

Thermopolis—
Freeman, G. N.

CANADA.

BRITISH COLUMBIA

Nelson—
Fowler, S. S.

Trail—
Batchelder, J. K.
Hamilton, E. H.
Lytle, L. B.

Vancouver—
Barwick, W. S.
Hayward, R. F.
McIntosh, D.

MANITOBA

East Kildonan—
Baker, L. R.

Winnipeg—
Armes, H. P.
Morden, G. W.

NOVA SCOTIA

New Glasgow—
Cantley, T.

ONTARIO

Kakabeka Falls—
Farrow, P. R.

Kingston—
Goodwin, L. F.
Goodwin, W. L.

Niagara Falls—
Arison, W. H.

Ojibway—
Baltzell, W. H.

Orangeville—
Deagle, L. M.

Orillia—
Tudehope, H. R.

Sandwich—
Walker, G. H. P.

St. Catharines—
Heitmann, E.

Sudbury—
Morin, H. A.

Thorold—
Atwood, F. C.

Toronto—
Bain, J. W.
Barrows, W. S.

ONTARIO—Continued

Toronto—Continued
Bennie, P. McN.
Boswell, W. O.
Burt-Gerrans, J. T.
Gaby, F. A.
Guess, G. A.
Hedstrom, E. S.
Kaemmerer, J. A.
Kenrick, F. B.
MacDougall, A. J.
Mathewson, E. P.
Miller, W. L.
Moffat, J. W.
Salisbury, E. F. W.

Welland—
Carnegie, E.
Eaton, I. C.
Guinther, John
Humbert, E. P.
Kelleher, James
Renton, W. L.
Turnbull, R.
Wilson, W. K.
Young, John

Windsor—
Henderson, E. G.
Vorce, L. D.

QUEBEC

Buckingham—
Hambley, F. J.

Chandler—
Hedin, Jos. E.

Montreal—
Davidson, T. R.
Hough, A.
Medbury, C. F.
Pascoe, C. F.
Randall, H. E., Jr.
Stansfield, A.

Ottawa—
Haanel, E.
Simpson, Louis

Quebec—
McKee, G. M.

Shawinigan Falls—
Acton, E. H.
Witherspoon, R. A.

Westmount—
Spencer, A. G.

CUBA.

Coahuila—
Berthier, U. H.

Mexico—
Hinton, Geo. B.

Isle of Pines—
Roberts, I. L.

SOUTH AMERICA.

BRAZIL

Rio de Janeiro—
De Medeiros, T. S. V.

S. Paulo—
Bowles, R. H.
De Souza, E.

Villa Nova de Lima—
Jones, H.
Wilder, F. L.

CHILE

Chuquicamata—
Dalmeida, J. A.
Jörgensen, E. L.
Krog, Karl M.
McClenahan, J. S.
Merrill, M. W.
Oldright, G. L.
Smith, F. T.
Wise, J. B.

SOUTH AMERICA—Continued.

CHILE—Continued

Rancagua—
Harper, H. A.
Stevens, W. L.

Santiago—
Antunez, V.
Cardoen, Remy
Diaz-Ossa, B.

CHILE—Continued

Valparaiso—
Hobsbawn, I. B.

PERU

Lima—
Boesch, J. E.
D'Ornellas, T. V.

AUSTRALASIA.

NEW SOUTH WALES

Broken Hill—
Gepp, H. W.

Port Kembla—
White, E. A.

Moonta Mines—
Hancock, H. L.

Sydney—
Du Faur, J. B.
Murphy, R. K.

NEW ZEALAND

Christ Church—
Stark, E. E.

Dunedin—
Shepherd, F. R.

QUEENSLAND

Brisbane—
Henderson, J. B.
Jackson, A. G.

Mt. Morgan—
Johns, M. J.

VICTORIA

Melbourne—
Gillies, P. M.

TASMANIA

Hobart—
Chappell, Wm. C.

Queenstown—
Sticht, R. C.

ASIA.

CHINA

Lincheng—
Kwang, K. Y.

INDIA

Mysore State—
Tate, J. V. E.

JAPAN

Echigo—
Nakamura, Yushichiro

Fuknoka—
Kaneko, K.
Tatsumi, Eiichi

Fukushimaken—
Nakashima, Shigemaro
Tada, Kozo

Kawasaki—
Matsushita, N.

Kobe—
Murahashi, S.
Tachihara, Jin
Tada, S.

Ishikawaken—
Iwai, Kyosuke

JAPAN—Continued

Korea—
Kawamura, Takeshi

Kyoto—
Nakasawa, Yoshio
Namba, M.
Nishikawa, Kikei
Yoshikawa, K.

Nagano—
Takahashi, Taketaro

Nikko—
Kaku, Juroku

Tajima—
Higashi, S.

Tokyo—
Gotoh, Issaku
Igarashi, T.
Ishikawa, Ichiro
Katsura, B.
Kameyama, Naoto
Kishi, K.
Masujima, B.
Nishida, Hirotaro
Nohara, T.
Watanabe, H.
Yamasaki, J.

AFRICA.

CAPE COLONY

Somerset, West—Quinan, K. B.

EUROPE.

AUSTRIA-HUNGARY

Brunn—
Babrowsky, G.

Treibach—
Fattinger, F.

Vienna—
Helfenstein, A.

BELGIUM

Brussels—
Cito, C. C.
Solvay, A.

Jemeppe—
Queneau, A. L. J.

Mons—
Henault, O-Dony.

Neerpelt—
Raeder, Bjorn

FRANCE

Paris—
deGeofroy, A.
Gall, H.
Keller, C. A.
Marie, C.
Ostheimer, J. W.

GERMANY

Berlin—
Finckh, Carl
Haber, Fritz

Bonn—
Bucherer, A. H.

Charlottenburg—
Engelhardt, V.
Reissenegger, H.

Clausthal—
Hoffmann, R.

Dresden—
Schrey, A.

Essen-Ruhr—
Goldschmidt, Hans

Frankfurt—
De Neufville, R.

Hamburg—
Arnemann, P.
Wohlwill, H.

Hanau—
Heraeus, H.

Karlsruhe—
Askenasy, P.

Leipzig—
Schall, C.

Luebeck—
Draeger, A. B.

Nurnberg—
Forchheimer, P. J.

Schwerte—
Amberg, Richard

GREAT BRITAIN

Birmingham—
Threlfall, R.

Irvine (Scotland)—
Walker, J. W.

GREAT BRITAIN—Continued

Liverpool—
Smith, A. T.

London—
Ashcroft, E. A.
Chalas, A.
Cornthwaite, Haydn
Cowper-Coles, S.
Goldstein, P.
McBerty, F. R.
Merz, C. H.
Oakden, W. E.
Ruthenberg, M.
Sprague, E. C.

Manchester—
Pring, J. N.

Middlesex—
Jacob, Arthur

Sheffield—
Cutts, V. O.
Dalton, A. C.
Fawcett, P.
Hadfield, R. A.
Klowman, Henning
Rowland, T.

Stafford—
Hill, Stafford

Walthamstow—
Wilder, F. L.

Widner—
Pritchard, D. A.

ITALY

Aguila—
Dapples, A.

Ligure—
Giolitti, F.

Milan—
Rossi, C.

Rome—
Catani, Remo
Chiaraviglio, D.

NORWAY

Bergen—
Turner, M. R.

Christiania—
Bjornson, E.
Bryn, K.
Collett, E.
Collett, Ove
Goldschmidt, Heinrich
Gronningsater, Anton
Hiorth, A.
Hiorth, F. V. L.
Hole, Ivar
Johansen, G. H.
Lenschow, H. H.
Loken, Rikard
Olsen, T. S.
Söderberg, C. W.
Tharaldsen, Filip
Thorne, C. A.
Torell, T. F.

Fredriksstad—
Moltkehansen, I. J.

Hardanger—
vonKrogh, Johan

EUROPE—Continued.

NORWAY—Continued

Odda—
Giersten, S.

Rjukan—
Bonnevie, H.

Stavanger—
Jacobsen, F.
Schubert, H. R.

Trondhjem—
Farup, P.

Vadheim—
Kolkin, T. L.

RUSSIA

Abo (Finland)—
Aminoff, G.

Petrograd—
Yatsevitch, M.

SPAIN

Huelva—
Shields, John

SWEDEN

Dannemora—
Leffler, J. A.

Falun—
Yngstrom, L.

Gottenburg—
Olsson, Henning

SWEDEN—Continued

Gullspang—
Hedlund, M.
Neumann, Wilhelm

Mansbo—
Carlson, B.

Stockholm—
Forssell, J.
Groenwall, Assar
Lilja, S. G.
Ljungh, Hjalmar
Tingberg, Otto

Trollhattan—
Berglund, E. S.
Olaussen, K. O. E.

Uddeholm—
Lindberg, S. C.

Vesteras—
Edstrom, J. S.

Westervik—
Tillberg, E. W.

SWITZERLAND

Basel—
Fichter-Bernoulli, F.

Geneva—
Gandillon, A.
Guye, P. A.
Lacroix, H.

Turgi—
Landolt, H.

An address delivered at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917.

THE PRODUCTION OF ELECTRICITY BY STEAM POWER

By Alex Dow.*

I came here tonight to keep a promise which was broken some six months ago. I was then called to Washington on some business of alleged national importance, which did not prove to be of great moment. These things are the troubles which beset us all in war times. To see so many people present at a war-time convention is a surprise and a great delight.

You are more interested in power delivered and available for your work than you are in the question of generating or producing power at the terminals of a dynamo-electric machine; therefore, it will be necessary for me to talk discursively, to get into questions of distribution and transmission, and to some extent into the utilization of power in order to get before you the ideas which I think you expect me to present, namely, the present status of the production of electric power from steam, as I understand that status.

I have not prepared a paper for you; I tried to avoid that sort of thing. It is one thing to talk to an audience on a subject fairly fresh and recent in your mind, and another thing to write a composition which will stand critical analysis in the office or the research laboratory afterwards. And I have an inherited national prejudice against reading an address anyhow.

You know the tale of the Scotch preacher whose early effort was a great failure, and he did not know why until he asked an old friend, who said, replying to the preacher's question of why he failed: "Robbie, there were three reasons: one was, you read it; the second was, you read it badly; and the third was, it was not worth reading."

I intend to avoid the first two reasons, and as to the third trouble, you may judge for yourselves later.

* President, Detroit Edison Co., Detroit.

Let me make the point again, that delivered power and generated power are two different factors. Generated power is the actual power produced at the terminals of your machine; the output is the amount of power delivered from your station into the trunk lines. The difference between the two represents uses in the power house, lighting, heating, excitation, the operation of auxiliaries, and all the minor losses incurred in any power-plant operation. The net power output is what the great majority of central-station men are concerned with. Then they have a third expression "cost of power delivered," and that is the amount of power delivered to consumer and presumably paid for. The confusion between these three is sometimes, I am sorry to say, intentional. Some persons have the habit of expressing their costs in generated units. In one instance I found a discrepancy between generated units and units delivered to the lines for distribution use, of the order of ten percent. And when that error was corrected the costs boasted of were not worth looking at twice. Costs said to be remarkable proved to be only good average practice.

The final figure, delivered power, varies again a great deal. In a close net-work, such as serving one big mill owning its own power plant, the difference between output and the actual delivery to the points of use may be eight or ten percent. In a complex system, covering a large area, and delivering power in different forms, the order of difference is more likely to be 20 percent and upwards. That is to say, if the customers' meters of a large central-station net-work account for 80 percent of the output from the power house, they are doing very well. That distribution efficiency is first-class practice.

There are many well-operated net-works, which, from obviously unavoidable conditions, do not have a distribution efficiency exceeding 72 or 73 percent. Commercially their practice is all right, but the difference between power generated and power delivered by the central-station systems to the customers' terminals is large enough to be an appreciable factor in the cost.

To come here and talk to this audience one should qualify as a teacher. Once in my lifetime I qualified as an expert witness, and I hope the Lord will forgive me for it. It was somewhere about 1870—I am quite as old as that—in 1870 I made my first

acquaintance with the inwardness of electric power. My text-book was one which I doubt if any one here would accept. It was Dr. Lardner's Museum of Science and Art. I read that very earnestly as a child, and therein I made the discovery that James Watt did not invent the steam engine. And I made a further discovery which many of my good friends have not yet discovered; which is that what he did invent is the separation of the condenser from the cylinder. Somehow it seems to me that every development of efficiency in steam power from James Watt's day to our own, has been in further separation.

The first division in the steam chamber was made by James Watt; the separation of the steam chamber into successive chambers, all the way from double to sextuple chambers, is within the memory of most of you here; and the establishment, instead of successive chambers, of a long gradient, as in the turbine, is a matter of the last fifteen years. We are now separating the functions of the condenser into condensation of steam and removal of air.

Therein—in separation—has lain every vital improvement in the steam engine. The others have been, not in physics, but in mechanism.

The next date in my memory is 1888—when I got acquainted with a certain 12 by 12 high-speed engine running 280 or 300 revolutions per minute, in Southern Illinois, in which, and in the electric system connected with it, my then employers had a fifty percent interest. And the problem then put to me, I being an alleged electrical engineer—I say "alleged" advisedly—was why if an engine of that type would produce on test a horsepower for every four pounds of coal, why that particular beast should require ten or twelve pounds. That involved a determination of what the engine was really doing, and it startled me to find that its consumption was in fact 17 pounds of coal per horsepower hour. There are a number of varieties of that engine, and it has many brothers today. You must blame the combination—of boiler, and engine, and then possibly throw in some of the human element. But when I got through I had a clear idea of what load-factor meant, and what cylinder condensation meant. I became familiar with, and contemptuous of, automatic cut-offs, and to think that throttling had some virtues. It had, and still has.

The Willans Law—the law that the steam used by any engine is a constant plus a variable, the variable being proportioned to the load, was announced about that period, and was one of the first recognitions of what actually went on in a high-speed engine. Willans (you may remember) divided his very successful engines into many chambers. He reduced the wetted surfaces; he produced a wonderful valve, and those engines of his are still continuing in service today, and they are very hard to beat in steam efficiency, especially if given a little superheat.

I find that vision of load factor had become a rule, a guiding law, to me, when in 1894 I found myself called upon to design a plant for a street-lighting load, a matter of 2,000 h. p., in four or five engine units, with, at night, a street-light load of 4,000 hours a year, shut down at the coming of daylight; and only a trifling load all day.

I deliberately selected a marine type steam engine for 160 to 170 pounds steam, at practically a fixed cut-off, although there was a governor of a sort; and I met the occasion of a day load, which ran from 150 h. p. down to 75 h. p. by the expedient of letting steam pressure run from 160 or 170 pounds down to 80 pounds on the boilers—down as low as it would go before I did any new firing. The result was it reduced the use of coal to four pounds per kilowatt hour, which would be equivalent to 2½ per indicated h. p.

In that design there was a point I would like to put before you, as it illustrates the cost of distribution. I and my associates deliberately chose series-wound dynamos of rather low efficiencies, down in the eighties, as against bigger dynamos of better types, because the problem had to be treated as a whole as one of cost of current actually delivered to the street lamps; and since the circuits ran into some fifty square miles of territory to light, it was highly desirable to do as we did and keep transmission losses down to seven or eight percent, instead of accepting higher power-house efficiencies and let the other losses run to 18 or 20 percent, which the higher power-house efficiencies would have predicated. Please notice the acceptance of the lower efficiency at the power house, to obtain maximum delivery at the point of use.

In 1908 I believe it was, I made my first personal, real contribution to the reduction of steam-power cost. Up to that time

I was merely developing the ideas of other men. Then I came to the conclusion that a large boiler would be perfectly safe and feasible; that a very large furnace was desirable for burning bituminous coal; that stokers should have certain characteristics; and I was able to get the financial support to build a power house designed for 56,000 kilowatts, in which the boilers and furnaces were of that type.

In 1912 two of these boilers were tested very liberally by Dr. Jacobus, and the results of those tests are in the text-books now. They established the very great importance of the correct adjustment of the furnace, and the stoker as a part of the furnace, to the proposed fuel and the proposed work. They also decided that the efficiency curve, of the boiler and furnace together, is a very flat curve; that a range (in this instance) from forty percent of the nominal rating to 200 percent of the nominal rating of the boiler, was entirely practicable. That upper limit has now been much exceeded. A design for long range is essentially a compromise design. Please do not overlook that.

Lastly, in 1914 and 1915, came the Connors Creek power house, which represented the best I knew, and which is the occasion why I speak to you tonight. The figures of the operating cost of that power house have been given to the public. They have been published at six months' periods, and by years, and as far as I am advised they have not been, for long periods and for a complete power house, thus far improved upon. I know of three power houses in operation and some others coming through, which should beat them eight or nine percent; but their figures have not been given out, and we are from time to time assured that the beating to which we are entitled has not yet been given to us.

The three Tables, I, II and III, are tabulations of the Connors Creek plant costs for three periods of twelve months with a six months' shift between periods. They thus cover two years. It is necessary to use figures for one year in such presentations because of the difference between summer and winter costs and because of occasional temporary aberrations of service, or of fuel, which would give exceptionally good or exceptionally poor showings for shorter periods. It will be noticed that the increased cost is almost entirely in the increased price of fuel. To a small extent it is due to uneven quality of fuel, because for the last nine

months we had to burn any kind of coal we could get, and a change from one fuel to another is always temporarily accompanied by poorer combustion. The other causes affecting costs are very decided increases in hourly rates of pay, the effect of which is cancelled to a good extent by increased output; and the fact that, during the Table III period, No. 2 turbine was running without its last row of nozzles and buckets, and its steam consumption was thereby increased some four percent.

Note that the stated output is kilowatt-hours metered to outgoing 23,000-volt trunk lines, also that the stated load factor is a customary central station load factor based upon the maximum half-hourly reading of output during the period.

Let me say this: Fuel cost is the one figure on which comparisons can be made, and fuel cost must be expressed in heat units to make the comparison. These Connors Creek figures show year in and year out an approximation of 20,000 heat units per kilowatt-hour with periods below and periods above it. In cases where they exceed the 20,000 figure, the excess is of the order of

TABLE I.

Connors Creek Plant.

Costs Per Kilowatt-Hour of *Net Output.*
Twelve Months Ending June 30, 1916.

Production Account	Total Expense	Expense per Kwh.
Operation:		
Superintendence	$ 16,841.40	0.013c.
Wages	54,308.36	0.043
Fuel	197,554.76	0.158
Water	10.00	...
Lubricants	1,416.60	0.001
Station supplies and expense	5,562.85	0.005
Maintenance:		
Station buildings	7,786.96	0.006
Steam equipment	23,826.70	0.019
Electrical equipment	3,299.80	0.003
Total	$310,607.43	0.248c.

Kwh. output	125,158,800 kwh.
Maximum demand (30 minute)	35,000 kw.
Average load	14,300 kw.
Load factor	0.409
Coal per kwh.—pounds	1.44
B. T. U. per kwh	19,700

Table II.

Connors Creek Plant.

Costs Per Kilowatt-Hour of *Net Output.*
Twelve Months Ending December 31, 1916.

Production Account	Total Expense	Expense per Kwh.
Operation:		
Superintendence	$ 20,521.67	0.013c.
Wages	68,477.62	0.042
Fuel	282,135.47	0.174
Water	10.00	...
Lubricants	1,055.23	0.001
Station supplies and expense	10,037.92	0.006
Maintenance:		
Station buildings	11,711.94	0.007
Steam equipment	26,670.32	0.016
Electrical equipment	4,394.92	0.003
Total	$425,015.09	0.262c.

Kwh. output	162,117,600 kwh.
Maximum demand (30 minute)	36,000 kw.
Average load	18,500 kw.
Load factor	0.514
Coal per kwh.—pounds	1.45
B. T. U. per kwh	19,800

Table III.

Connors Creek Plant.

Costs Per Kilowatt-Hour of *Net Output.*
Twelve Months Ending June 30, 1917.

Production Account	Total Expense	Expense per Kwh.
Operation:		
Superintendence	$ 21,756.67	0.010c.
Wages	98,710.84	0.047
Fuel	503,447.63	0.240
Water	.51	...
Lubricants	1,193.73	0.001
Station supplies and expense	11,096.28	0.005
Maintenance:		
Station buildings	22,174.19	0.011
Steam equipment	40,353.83	0.019
Electrical equipment	2,939.75	0.001
Total	$701,573.43	0.334c

Kwh. output	210,039,700 kwh.
Maximum demand (30 minute)	50,000 kw.
Average load	23,900 kw.
Load factor	0.478
Coal per kwh.—pounds	1.52
B. T. U. per kwh	20,040

1½ or 2 percent, and when they get below it, it is at very favorable seasons of the year. They are the best which can be done with the present limitations of that power house.

The house is of a design, and a selection of machinery, of the years 1913 and 1914. It is not completed. When completed it will do a little better. The reduction of losses by radiation from the boilers proper is so extreme that the boiler room must be heated in winter for the firemen. The turbines are essentially 12 lb. per kilowatt-hour machines, and are habitually operated at their best points, say 11½ on test conditions; but we do not rate them at that.

The particularly interesting feature is the arrangement made for getting the most out of the steam used by auxiliaries; and here begins what I might call Chapter 1 of Instruction—because so far I have only given you historic classification—as follows:

Heat Balance: You shall not throw heat overboard beyond what you cannot save, and you shall get as much power out of all the steam you make as is possible.

Now, if you will turn your memory back you will recall that feed-pumps and condenser pumps and auxiliaries of all kinds were once upon a time habitually driven from the main engine, and therefore they had the efficiency of the main engine. Marine practice, until recently, was exactly that way, and thereby rose a compromise, due to the limited spaces and weights in marine practice, of running at 24 to 25 inches vacuum; because experience showed that to put the feed-water into boilers at temperature corresponding thereto was more efficient than to get better vacuum at the expense of colder feed-water. Colder feed-water meant you were pumping too much heat overboard; and that loss overboard is the one hopeless proposition today. You will remember that something like eight-tenths of the heat units are hopelessly lost in the condensing system; and therefore any heat process which uses electric heat originating in fuel carries a heavy initial handicap.

A mighty poor combustion of fuel—be it coal or oil or natural gas—should give fifty percent efficiency of heat transfer into the work. I think the domestic gas range gives between 35 and 50 percent efficiency when operated by a reasonably careful cook, who does not let the gas burn all night through neglect to shut it

off when through using it; and an efficiency of fifty percent of the delivery of the heat to the work from combustion, in a process of that kind, should be made every time, and higher efficiencies should be practicable. On the other hand, by our present methods you will never get higher efficiencies than that corresponding to the possible 100 percent efficiency of the transformation of electricity into heat, multiplied by the possible 20 percent efficiency of the transformation of fuel energy into electricity. Therefore, in electrical heating units, you use electricity because of electricity's other merits—not for its thermal efficiency from fuel to work. And its large use proves that wise men are willing to pay for those other merits, and that fuel economy is not the only thing to consider in industry.

Put me down as saying that where electric heating, electric steel making, electric reduction of ores—that when these things are used and the origin of the electricity is in fuel, you are absolutely applying that heat wastefully considered as an application of heat, and the wastefulness is hopeless. The loss overboard in the condensing water is the big loss. All other losses, such as stack losses and radiant losses from boilers and engines, are minor in comparison.

The losses in auxiliaries have been very great. You will recall that the next style of auxiliaries was in the use of separate direct-acting steam pumps, which were popular improvements in pumping devices; and we had the theory propounded that inasmuch as these were always operated with a closed heat cycle, the steam used in those auxiliaries was not a loss, but the heat therein was wholly returned to the system. That was true, but the heat was returned without removing anything like the amount of power which should have been removed.

The Connors Creek auxiliary plan, which as far as I am aware was not preceded by and certainly not taken from any earlier design, but was one of my own sketches, made in 1912, is to operate the auxiliaries by electric drive from a separate turbo generator, exhausting into a condenser whose temperature is controlled according to the requirements of the feed-water. That is to say, you run a separate electric generator of comparatively small size, and from that you drive your auxiliaries (except a steam turbine feed pump) and the exhaust from the turbine of

that generator goes to heat your feed-water to the economical temperature. That economical temperature being settled by other considerations, you get from the steam which goes to supply your auxiliaries, all of the possible power and likewise all of the heat; and your auxiliaries have the great convenience of the electric drive.

At Connors Creek we have a method which is capable of various and numerous changes. In our last sketch the feed-pump is also made electrical, with a standby steam feed-pump to meet occasional conditions. The combinations possible are legion. I could talk to a mechanically interested audience at least three hours on this subject. Right there let me say that *all-steam* drive of auxiliaries must be wrong; and *all-electric* drive usually is wrong. The correct choice lies between the two.

Let me say about Connors Creek, there is no economizer there; that is a commercial proposition. The use of the economizer would unquestionably save four percent of the heat units; and commercially, under the conditions of coal cost as they were two or three years ago, it would not pay. At the present time it would probably pay if the thing could be "wished into place," and the problems required to be solved in the all-steel economizer could be solved with despatch. If coal stays at the present ordained figure—not to speak of the actual figures we are paying—the economizer will be warranted and we have saved space for it. Under the conditions of the past the economizer would not have justified its existence. If put in, the economizer would better the economy four percent; then the turbines could be replaced by similar machines four to five percent more efficient; and with the heat balance betterments we can see, we would pick up one or one and a half more percent. That is to say, we could reduce the present 20,000 heat units per kilowatt-hour to 18,000. That is an entirely practicable figure at present, without going into new design. There are certain ways whereby a still lower annual heat cost could be obtained in practice, and they are commercial, such as improved load factor.

Let me say also there is a difference between summer and winter efficiency, as in winter you have a much better vacuum. The colder water of our lakes and rivers in the winter months means that a better vacuum is practicable. On the other hand, if you

over-cool your condensate you are losing too much heat overboard. And the winter radiation losses from a power house, built as a power house ordinarily is, and not as a refrigerator plant might be, are a decided loss.

Now the Total Cost is the fixed cost plus operating. The division sometimes made is into standing and running costs—the standing costs being those which make the station ready for service, and the running costs being the added cost due to service or output. You get there an equation like Willans Law. Let me say this: the fixed element is so very large that at poor load factors it is controlling. With some of you the load factor is high; with others it is inclined to be low. The electrochemical load factor is generally recognized as specially high; it is of the order of 90 percent of what would be obtained if the maximum hours, that is 8,760 hours, of maximum demand were obtained during the year. You gentlemen are in the habit of telling us you have 100 percent load factor. Ninety percent is going strong, and figures in the eighties are more frequent. On the other hand, a mixed service is doing well if it shows 40 percent. Which, expressed otherwise, would be, that if that station could operate at full load for four-tenths of the time its output would be the same as it is, at the loads available, for the whole 8,760 hours of the year.

Any central station with the load factor above 40 percent is doing well, indeed. Below that is common enough. The load factor of residence lighting is of the order of eight percent; which means that the costs for residential lighting service are nearly all fixed charges. You can almost forget running charges on residence business. Since it is the custom to charge according to kilowatt-hours used, the rate per kilowatt-hour for residence service is much higher. Did we charge according to the rate forms used for electrochemical service, the difference would be seen to turn on that one point of load factor. Residence service uses the equipment about eight-hundredths of the time that electrochemical service might use it.

Now, as to the Capital Cost, a fixed or first element—the capital cost of the Connors Creek plant, for the first units, is between $50.00 and $60.00 per kilowatt. If you buy land for 300,000 kilowatts you must pro-rate that over the proposed investment or in-

stallation; and you put in railroad tracks, and dike out lakes, and put in all the facilities, and offices, when you build your first units of a big plant, all of these cause the unit cost to go high; therefore the variation is between $50.00 and $60.00. But the crazy prices of the last two years would make the cost go between $90.00 and $110.00 per kilowatt. One hundred and eighty percent is just about the cost of such construction now as compared to the cost we considered normal before the world went crazy.

Now, money for that sort of business formerly could be had at rates which did not very much vary.

Taxes on a public-service plant are assessed on a liberal basis. We are very honest, we public-service people. We always confess to a liberal and honest basis for taxation. Now that is no slight matter. Communities which in the past have favored the manufacturer with a low estimate on his plant, for taxation purposes, will assess a public-service corporation up to the eyes; and let me say that taxes in the last year required us in Detroit to earn $1,600.00 a day, to pay taxes, every day, Sundays included.

The cost of money, plus the depreciation resort, plus the taxes means that the central station must figure on a return never less than 12 percent, and sometimes exceeding 15 percent. So your first figure, based on the central-station method of obtaining your power supply, is to figure 12 to 15 percent as a figure on an investment per kilowatt, which was between $50.00 and $60.00 and which now is between $90.00 and $110.00.

Let me say a word here about Depreciation: Now, the depreciation conditions are really affected by an electrochemical load. Depreciation, save and except "obsolescence," is higher with higher load factor. Depreciation will be some function of use, and a high load factor will obviously mean a higher rate of depreciation. That should be remembered in figuring costs of any purely electrochemical plant.

Now, assume we have the problem put to us by some of your members, that you want a power plant, the object being low total cost of power, and you have some choice of location. The first thing to consider is how the location may affect the power plant; and that leads to discussion of those non-engineering problems I spoke of earlier.

With electrochemical industries, of course, you must consider

your raw material and your markets. You must have good shipping points; you must have fairly good labor conditions, etc. These conditions would have a large influence in determining the location; and if you leave us free to transmit the power over from a satisfactory location to the power plant—if that condition be in the premises of this discussion, we can forget these considerations for the present and consider one location against another as reflected in design.

Take Item No. 1—the fuel cost. If the load factor is of the order of 90 percent, fuel cost will be the most important thing to consider. In connection with fuel cost you must consider the certainty of the supply—that it will not be interrupted.

Then you must have plenty of water. The quantity of water used by a big steam turbine plant is something you do not conceive. I know, as every engineer does, the per capita water consumption of a mixed residential and commercial community such as Detroit. I also know the amount of water required to condense the steam for our big turbines. A plant of 100,000 kilowatts, operating at 50 percent load factor, will require to pass through its condensers approximately the same amount of water as will be used by a mixed residential, industrial and commercial community having a population of 750,000 people. In other words, my power plants take up from the river, heat 10 or 15 degrees, and reject to the river again, more water than is pumped for the whole Detroit community. Therefore, the water supply is the next thing to consider after a good supply of fuel. You must locate on navigable rivers or streams, or on the Great Lakes. Cooling towers are devices you must forswear if you are working for maximum efficiency. One of the appropriate places for a cooling tower is in the dry Southwest where the rapid evaporation in an exceedingly dry atmosphere cools the water sufficiently, and the night chill gives you a fair start the next morning. To locate a big plant on a country duck pond is foolishness.

The next consideration of importance is the labor conditions. Power-house labor is changing its character very much. There was a time when the power house got all the incipient geniuses; that was a mistake. But the day has come for brains, and they are being secured. Today the power-house employes expect good pay and good environment. These employes will jump in and do

a dirty job and do it handsomely, but they are men who appreciate bathing facilities. I recall a tale of an old-time engineer who discussed the showers which were being placed in the plant. He said he took one of them showers every Saturday whether he itched or not. But the engineer now gets his shower when he goes off watch, and sometimes he calls his relief and takes a shower while his turn is on. You must supply first-class working conditions, both inside and outside. These men expect their outside, off-work conditions to be such as they can pleasantly live in when they are off watch, where the older men can bring up their families and the younger members can hope to persuade "the only girl" to come.

And, remember, the right kind of labor is a big item. You will have to build, control and sanitate your place, if you locate away from a pleasant community.

If you locate on a place where people consider the outer appearance you must employ an architect. I can think of three power houses where the outside appearance is a credit to the public spirit of the board of directors, while the inside is a libel upon the architect. Foundations will run to five dollars per kilowatt when you locate in a marsh with rock 100 or 150 feet down, and you must pile or go down to that rock; or if you locate on the Ohio River where you have 70 feet of rise and fall, there you have another serious problem of foundations.

These things balance up. Low fuel cost can be offset by poor water supply. It is better to go a little further away from the coal to get better car service and so on. The next thing is whether you will locate close to your load, or will transmit.

Now, transmission is expensive. It has a very definite measure as to its capacity and a high figure as to its unit cost.

The right-of-way of your transmission lines is an item of high cost. The line loss may be a high cost. Some interesting figures were obtained when I was asked to furnish the comparative American costs of electric transmission of energy from coal mines against railroad movement of coal, for comparison with possible English costs, with an assumed line loss of 15 percent. The preliminary result of the comparison is that England cannot move energy by freight train as cheaply as by electric transmission lines, while American freight trains hauling short tons of coal at some-

thing like one-third of a cent per mile, can move it more cheaply than the transmission lines. There you see how you must balance one thing against the other.

Again transmissions are not utterly reliable. This was a year notable for lightning, when lightning was painful and frequent and free. We have such things as lightning arresters and they take care of secondary surges; but when you get a real direct stroke of lightning you go around with a shovel and clean up. Let the temperature be 30 degrees below zero, and the electric stress be hundreds of kilowatts; and then change in due course to our hottest summer weather. The porcelain insulators are supposed to adjust themselves to all of these conditions, while placed inside of a rigid metal cap. Well, there are limits to human beings and there are limits to the elements which constitute these strings of porcelain insulators. Thus you will find that lines which behaved splendidly for seven or eight years after that time behave miserably. And the cost of changing insulator strings is a very considerable item. There are three to a tower and at ten dollars each makes thirty dollars for each tower, and in a hundred-mile line that is an item. Then do not forget cyclones.

You must balance permissible unreliability against costs. If you are generating by steam power it is probable that your big center power plant will be *per se,* more reliable than any plant you can install on your own premises; but the intervening link may change the balancing conditions. You must consider whether you want the continuity of your chemical process to hinge upon the unavoidable conditions which I pointed out as inherent in the transmission line; and you must balance better locations for the power plant against line losses assumed at 15 percent. That is high. You may spend money and cut it down.

The distribution loss I spoke of earlier is of the order of 20 percent total. I have got here some figures from a big system. The total distribution loss is 18.7 percent; of that 5.6 percent is lost in the line, three percent is lost in the sub-station transformers; loss in the synchronous condensers—mighty few of them—is one-tenth of one percent; the conversion loss in changing from alternate to direct current is 4.7 percent; storage battery loss is of the same order as synchronous condenser loss; and then there are distribution losses proper, line transformers, meter shunts,

and slow meters to make up the difference. It may be considered absurd to put "slow meters" in as loss, but you have the alternative—you can keep them just a little fast or just a little slow; and you will keep them a shade slow when you have lived with them as long as I have.

So far as you are concerned I think the question of location versus transmission turns not on the question of reliability or unreliability, as duplicate lines could alter that; it turns on your own problems of materials and markets. The answer is, if the power cost controls, you must go where you can make power cheaply; otherwise, you must consider your raw materials and markets for your products and then make the best adjustment you can between reliability of transmission, the cheap power location, and your own conditions. It would be possible, I think, and I have said so to some of your members, to choose one of a considerable number of centers where a good adjustment could be made between all these conditions, and then the conditions of plant design would be predicated very closely.

As to generating costs with present practice, 19,000 heat units are entirely practicable. The other operating costs are of the order of one-tenth of a cent per kilowatt hour. You can figure the cost of your 19,000 heat units, plus one-tenth cent—and you might note that 17,000 heat units is possible, without any radical departure from present methods or materials.

What is the possibility of higher steam pressures? I speak of pressure above our present 250 to 300 pounds. If you get to consideration of the fact that you are working with high tangential velocities in the turbines, and talking of pressures of 300 to 400 pounds per square inch, and temperatures of 700 Fahrenheit and upward; if you consider that these pressures and velocities and superheats and temperatures are all present—you will realize that the selection and production of suitable materials of construction is a problem. I am told no steel manufacturer will guarantee analysis or qualities of steel at the present time. If that is true the ideal turbine will have for the present to be a thing to be dreamed of and not built. The technical difficulties are solely those of materials. The commercial reasons are likely to keep us under 300 pounds steam pressure and under 700 Fahrenheit, for total steam temperature, for some time to come.

The history of the things which already have been tried to get beyond that stage is unending. Some of us are now using high-carbon boiler tube because we punish soft steel too much already. The stories of warped castings and shrink-fits that did not stay shrunk, and erosion of blading, etc., are endless—and time flies.

As to Gas Fuel: Yes, there is a chance for betterment of economy in gas fuel. You can control your mixture; lose no carbon in your ashes, and have higher heat transmission values. But gas as a commercial proposition, in almost all of the United States, is entirely dependent for its cost upon the by-product market; and that (as you know) is anybody's guess. In the meantime gas producers, as an addition to boilers, cost too much. No combination in present or recent conditions can be made that will allow artificial gas fuel to be used in steam boilers; natural gas is now too valuable; and blast furnace gas has its uses in its own habitat.

The gas engine has its place. At present the large steam turbine has it beaten for general uses. With free furnace gas or natural gas, the gas engine has its commercial merits. But even in the blast furnace business, the steam turbine, with the turbo-blower, has possibilities of displacing the gas-blowing engine. I am told that so long as the intermittent blast of the big blowing engine tends to prevent hanging, they do not want centrifugal blowers. That may be a good practice point, and may be controlling.

Pulverized coal must be considered in any new design. It is first class now in big locomotives because of the impossibility of firing by hand the present big machines. There is a better control of the air mixture; and you cut off your fuel, and that is the end of it. It may permit the use in power plants of coals which now work very badly in stokers. Please remember that coal is not always coal; it is sometimes an invention of the devil, which should be called by another name. Coal that works into a kind of molasses candy is an exasperation, and not a fuel. Coal that fluxes down your furnace walls, and coal that must have acid furnace brick or basic furnace brick—the coal for one kind of fire brick may be the worst kind of coal for another kind—these may be dealt with by pulverization.

Now, the whole question of pulverized coal turns upon the

point that it may make coals useful, which with stokers of the present time are not useful, and thereby may reduce total power cost. The big point is to adapt the plant design to the fuel you are going to use.

Now, in closing: It is not good to prophesy, but I know some of you expect to hear from me whether I anticipate any great change in the art. I do not, just now. Dr. Lardner went on record as saying no steamship could carry enough coal to make a voyage across the Atlantic. That was accepted in the thirties, while in the forties there was a steamship line established. Therefore, all I can say is—there is not in sight any great change, *unless* we can find the materials to take us into much higher temperatures. Boiler and furnace efficiencies are now between 80 and 90 percent, including economizers, and we need not expect much change there. The remaining losses are commercial losses which cannot be saved profitably; such as the losses necessary to the stack to give you the necessary draft; or are like the big losses of heat thrown overboard, they seem to be inherent in steam. There may be a new cycle produced which will save all this. There is a man waiting for me to come home, ready now to produce that cycle—he says. I hope he has it.

I see also that the freeing of unlimited power is to be investigated by a national appropriation. I do not think that development will be an improvement in the direction we discuss tonight, in the making of electricity by steam power.

I have not told you all I know, but I have told you all I can crowd into a reasonable time, with a bad start.

DISCUSSION.

F. A. Lidbury[1]: In an address to this Society about eighteen months ago, in Washington, Mr. Lawrence Addicks expressed the view that the time had come when the electrochemical industries should cease to regard themselves as dependent on water power in this country, since, according to the figures which he mentioned, the recent advances in steam-power engineering had brought the costs of steam-power production to a point at which

[1] Works Manager, Oldbury Electrochemical Co., Niagara Falls, N. Y.

they approximated power prices at Niagara. His views and figures were not uncontradicted at the time, and might not have resulted in particular attention being given to this question by the Society had it not been for the fact that they came at a time when the electrochemical industries were suffering from a power shortage which was even then serious and which has since become a national menace. Under these circumstances a number of the Niagara members decided to look into the matter and see what could be done, as the situation seemed to be one which would not permit any stone to be left unturned.

We were extremely fortunate in meeting, in this respect, with that remarkable spirit of co-operation on the part of the Detroit Edison Company which has been so well exhibited in what Mr. Dow has said tonight. Mr. Dow, who was unable to present his views to the Society at the last meeting, was kind enough to come over to Niagara Falls and talk to us very freely, openly and frankly on the subject; but the result of this conference was, I am sorry to say, that most of us lost interest in steam power under present conditions. It seemed to be a matter of general agreement that if we could revert to pre-war conditions the matter would be worthy not only of general but of serious detailed consideration. However, with things as they are, with prices as they are, and particularly with the price of coal threatening to remain for an indefinite period at a previously unheard of figure, it would scarcely be possible to go into the generation of power from steam on a considerable scale at all, and it would be impossible to go into it at the present time on any basis that could be considered, for this purpose, commercial. In other words, we jumped at the idea of steam under improved modern conditions, and when we found what it meant under present circumstances, we jumped back as rapidly as possible.

Tendencies can always be checked by facts, and the facts in recent months indicate little or no tendency on the part of electrochemical industries to depart from water power. What we see today is just a continuation of the process which we have watched for a number of years proceeding at an increasing rate, and which as far as we can see at present will continue to proceed; and that is the expatriation on account of power shortage of industries which are essential for the needs of the country.

You all heard Mr. Randall's paper this morning, in regard to water power at Shawinigan; and now that the activities of the Hydro-Electric Commission of Ontario have resulted in a shortage of power at Niagara Falls, Ont., it appears that Shawinigan is offering the most favorable ground in Canada for the extension of those industries throttled at Niagara Falls, N. Y., and as you will observe, these industries are already beginning to move there, as formerly they moved to the Niagara district of Ontario. I should not be surprised to find in the next few years a healthy class of Niagara alumni at Shawinigan!

Now there is a national aspect to this matter. From the broad point of view it is very doubtful to what extent the country should permit the emigration of industries which are as fundamental as any group. And yet we have apparently got to let these industries go, as far as their growth and development goes, or we have got to treat the matter politically and decide that they must be kept in the country by hook or by crook, at whatever prices steam power might permit them to produce their goods, by the imposition of tariffs sufficiently heavy to be prohibitive. Or we might adopt the most sensible course, to permit and to insist on and to enforce the thoroughgoing utilization of our natural water power resources, and in particular of Niagara, a course of action which could solve this whole question inside of a few years.

We have been talking of improvement in this matter four or five years, and even longer. We have had a respectful hearing in our own Society. Outside we have been treated as cranks or as malefactors. The result is that this country is faced with an alarming condition with respect to its productive powers for a tremendous number of articles which are fundamentally necessary for warfare.

Nobody realizes at this time the extent and the seriousness of this matter. Nobody realizes it because there does not exist even today in the departments at Washington any clear idea of the quantities of materials and supplies which will be necessary, the production of which will depend upon the electrochemical industries of this country. We shall have a demonstration of the results of the policy which this country has developed in regard to water power legislation, and the only hope we can express is that when matters do come to a sufficiently clear indication of the

mess we are in, we will put ourselves in such a position that the same thing cannot occur again.

Before we adjourn, Mr. President, I move that we give a rising vote of thanks to Mr. Dow for coming here tonight and telling us in this masterly address the valuable things he has. There has always been need for co-operation and understanding between the electrochemist and the power man. Mr. Dow has not only combined willingness to co-operate with an understanding of the situation, but has added to these a broad viewpoint peculiarly his own.

C. T. HENDERSON: I take pleasure in seconding the motion of Mr. Lidbury, and in so doing would say that if Mr. Dow keeps on, it will be but a short time before we are able to compete with any water power in the country.

[The vote of thanks was passed unanimously.]

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

SHAWINIGAN AND ITS ELECTROCHEMICAL INDUSTRIES

By H. E. Randall.*

[Abstract.]

A description of the location, climate, power supply, present electrochemical industries, sources of supply of raw materials, shipping facilities, manufacturing conditions, and labor market, of the Shawinigan district, Province of Quebec, Canada. With 500,000 horsepower installed, of which 100,000 is available for new plants at once, and 600,000 available waterpower as yet untouched, and situated at 20 miles from tidewater on the St. Lawrence River, which is open to ocean navigation six months per year, Shawinigan is attracting very favorable attention as the electrochemical center of Canada.

LOCATION AND ORIGIN.

Shawinigan is located in the Province of Quebec, Canada (Plate 1), midway between Montreal and Quebec City, and derives its name from the Indian word indicating the resemblance between their beautiful quill and bead work and the great Falls of the St. Maurice River. (Plate 4.)

The town is of comparatively recent origin, and until 1898 its entire population lived in one house. Then came the original power development, soon after, the Canadian Northern Railway, then the Pittsburgh Reduction Co., then the Canadian Pacific Railway, and soon the wilderness of '98 became a thriving, progressive industrial city, with a population of 12,000 people, and manufactured products reaching the $20,000,000 mark in 1916.

Shawinigan, with its two railroads, is within easy reach of the

* Electrochemical Engineer, The Shawinigan Water and Power Co., Montreal.

larger cities of the continent, being three and one-half hours from Montreal and about 15 hours from New York, Boston, Buffalo and Toronto. About six trains a day are available to each of these points.

Shawinigan lies about 20 miles back from Three Rivers, where the St. Maurice River enters the St. Lawrence River. It is on the improved road system of the Province of Quebec and is thus easily accessible to motorists.

CLIMATE.

The St. Lawrence Valley being tempered by the large mass of water moving from the Great Lakes to the sea has a very steady and pleasant climate. The lower St. Maurice Valley (Shawinigan district) has a mean annual temperature of 45° F., and the mean temperature in the winter period is about 20° F. In summer the mercury climbs to above 90° F. for a few weeks, and in winter occasionally drops to —25° F. The annual rainfall is about 36 inches (91 cm.), of which 10 inches (25 cm.) comes as snow and largely remains throughout the winter period.

POWER SUPPLY.

The St. Maurice River, whose drainage area is upwards of 20,000 square miles (55,000 sq. km.) has a fall of 150 feet (45 m.) at Shawinigan. This fall has been utilized in the four power houses shown on Plate 7, which now have an installed capacity of 196,000 H.P. The latest power house at this development has five 20,000 H.P. units, and when constructed in 1910 represented a very big advance in hydro-electric practice. (Plate 5).

Nine miles above Shawinigan the St. Maurice River falls 85 feet (25 m.) and there is located another power house (Plate 8) with an installed capacity of 120,000 H.P. (Plate 6). The plans for this power house called for 60,000 H.P. of additional capacity, and during construction arrangements were made for the quick installation of this capacity, so that when needed it can probably be installed as fast as the load develops. This power house operates in parallel with the Shawinigan Power Houses, and it supplies the Shawinigan industries as well as feeding the general distribution system of the Shawinigan Company (Plate 11).

FIG. 4.

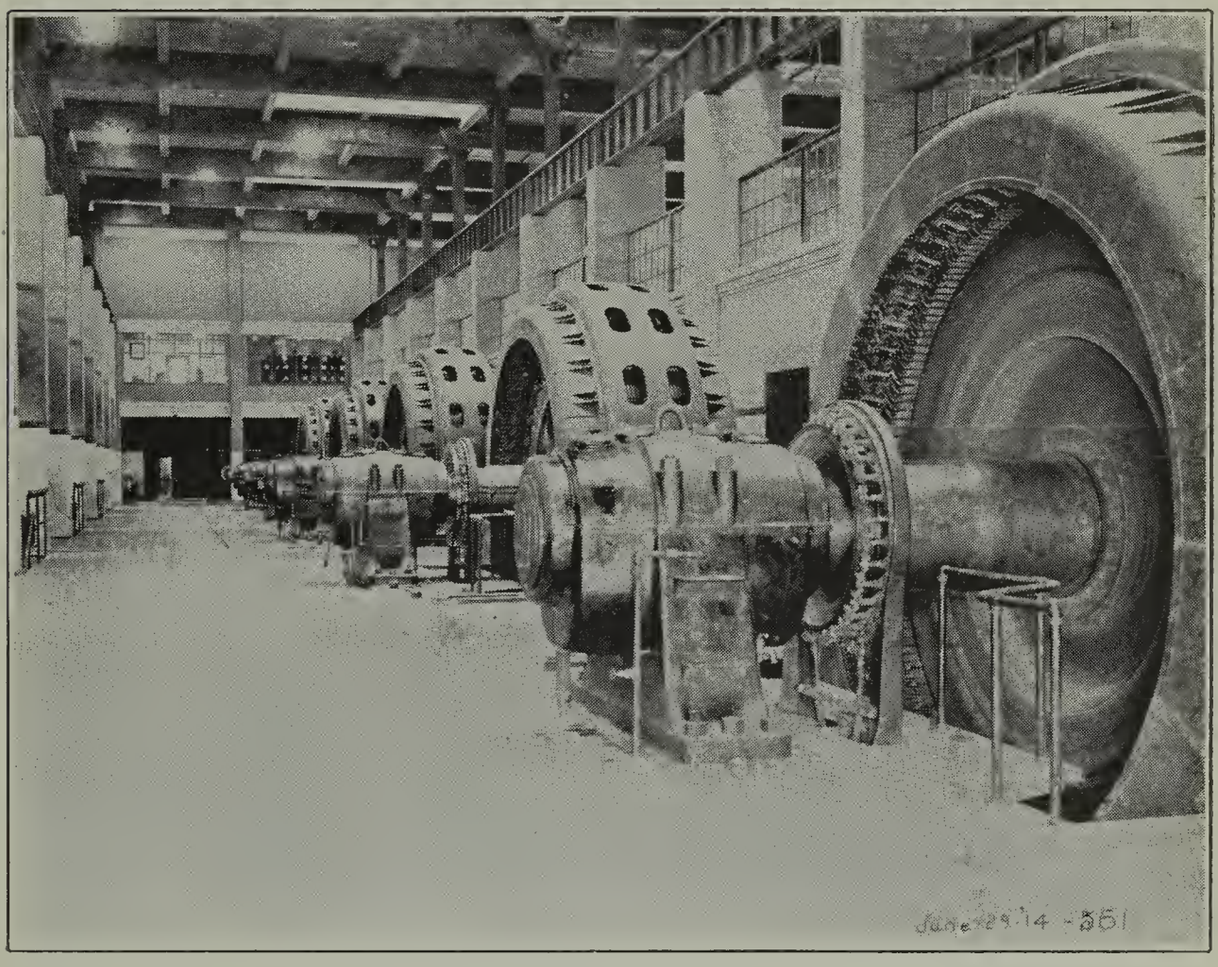
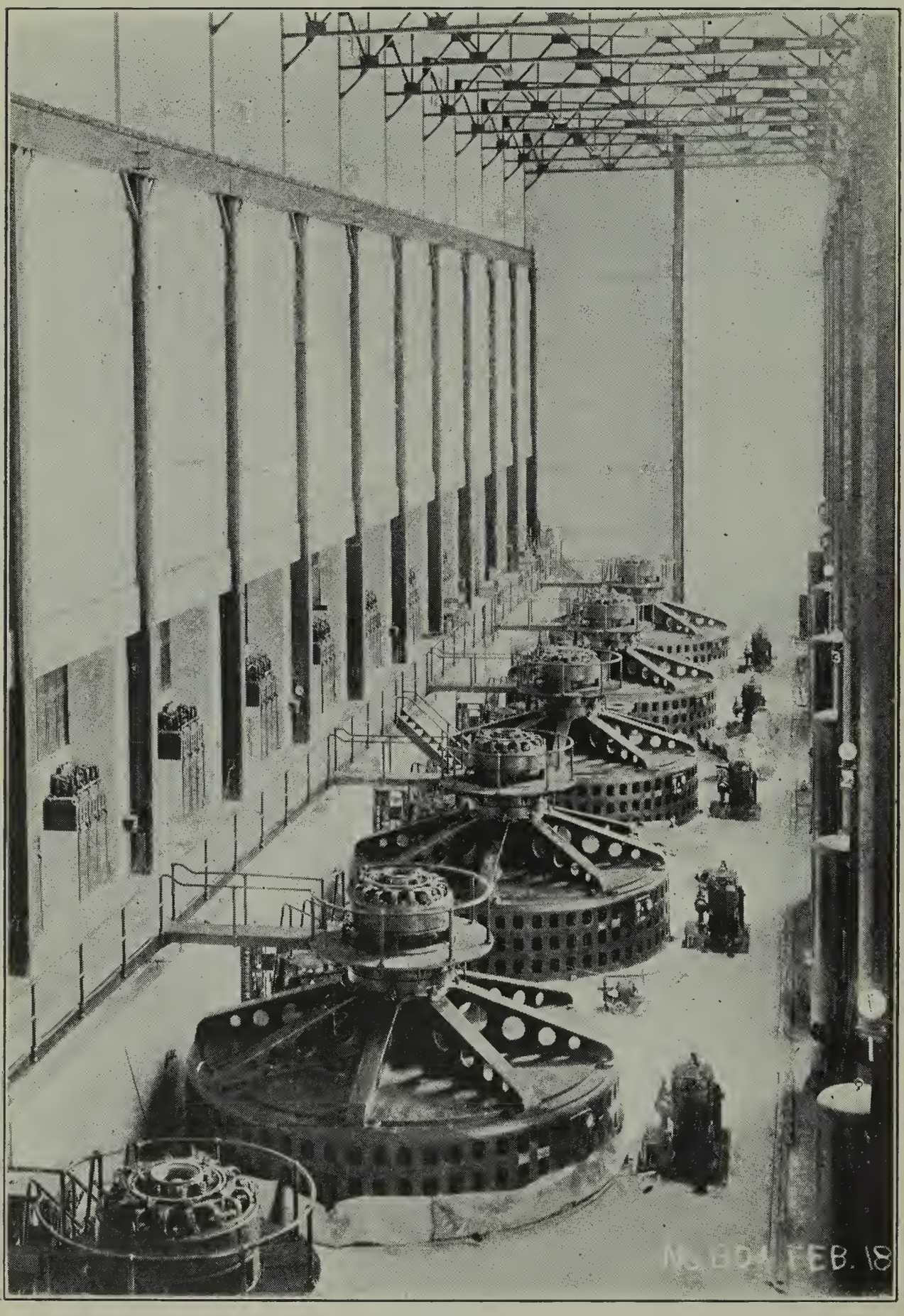


FIG. 5.



FEB. 18

FIG. 6.

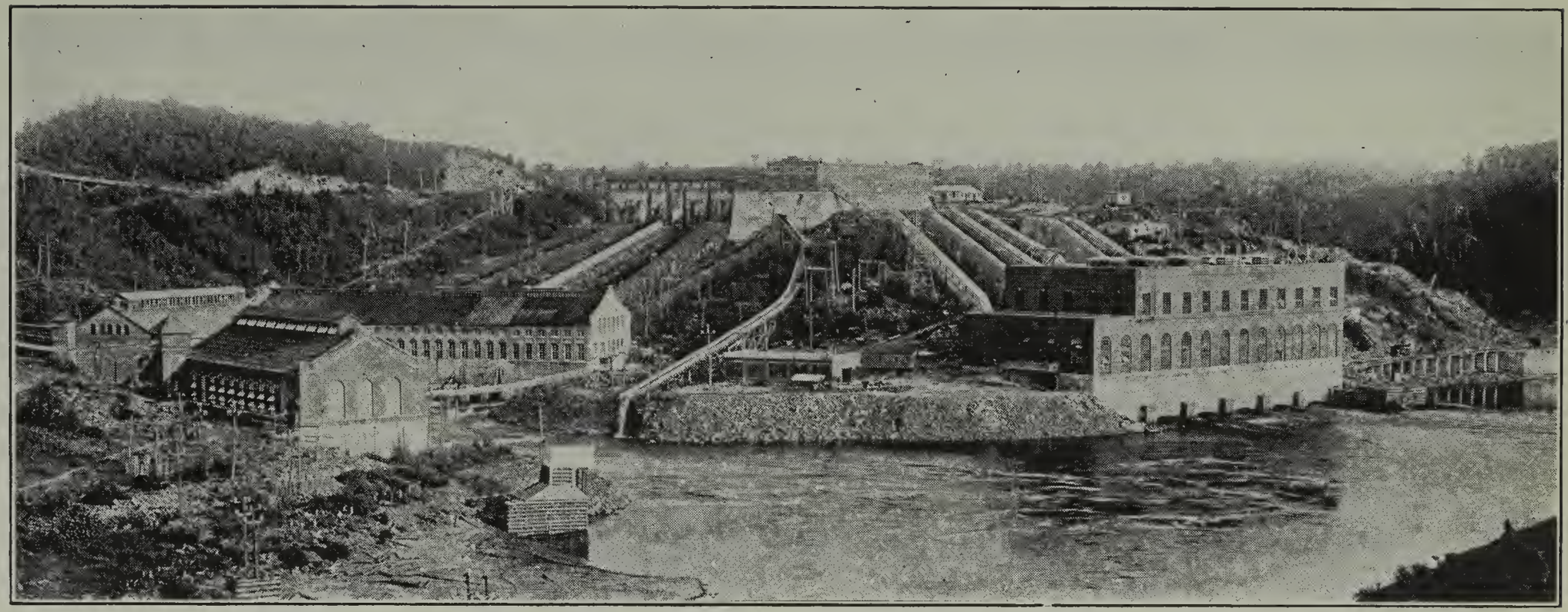


FIG. 7.

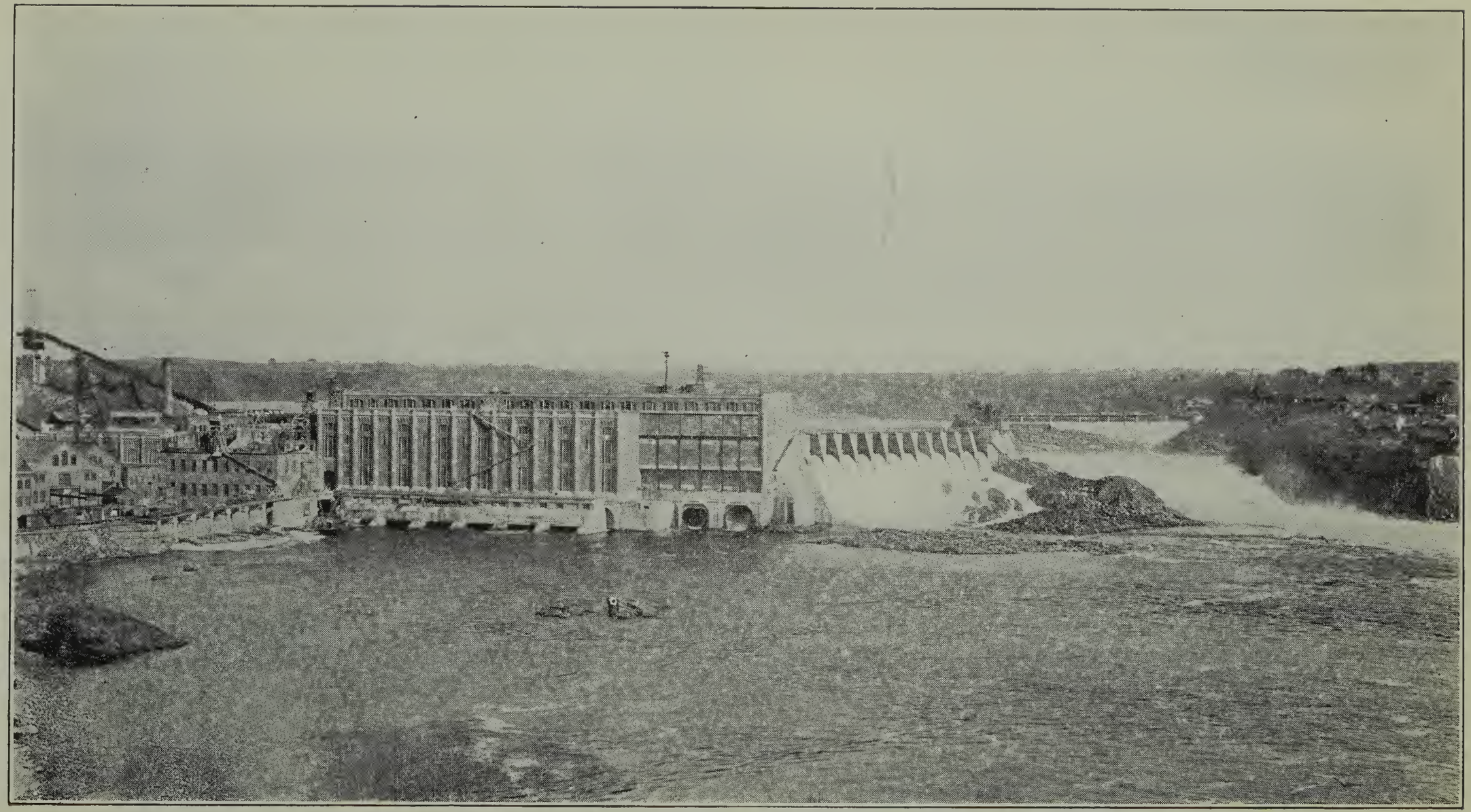


FIG. 3.

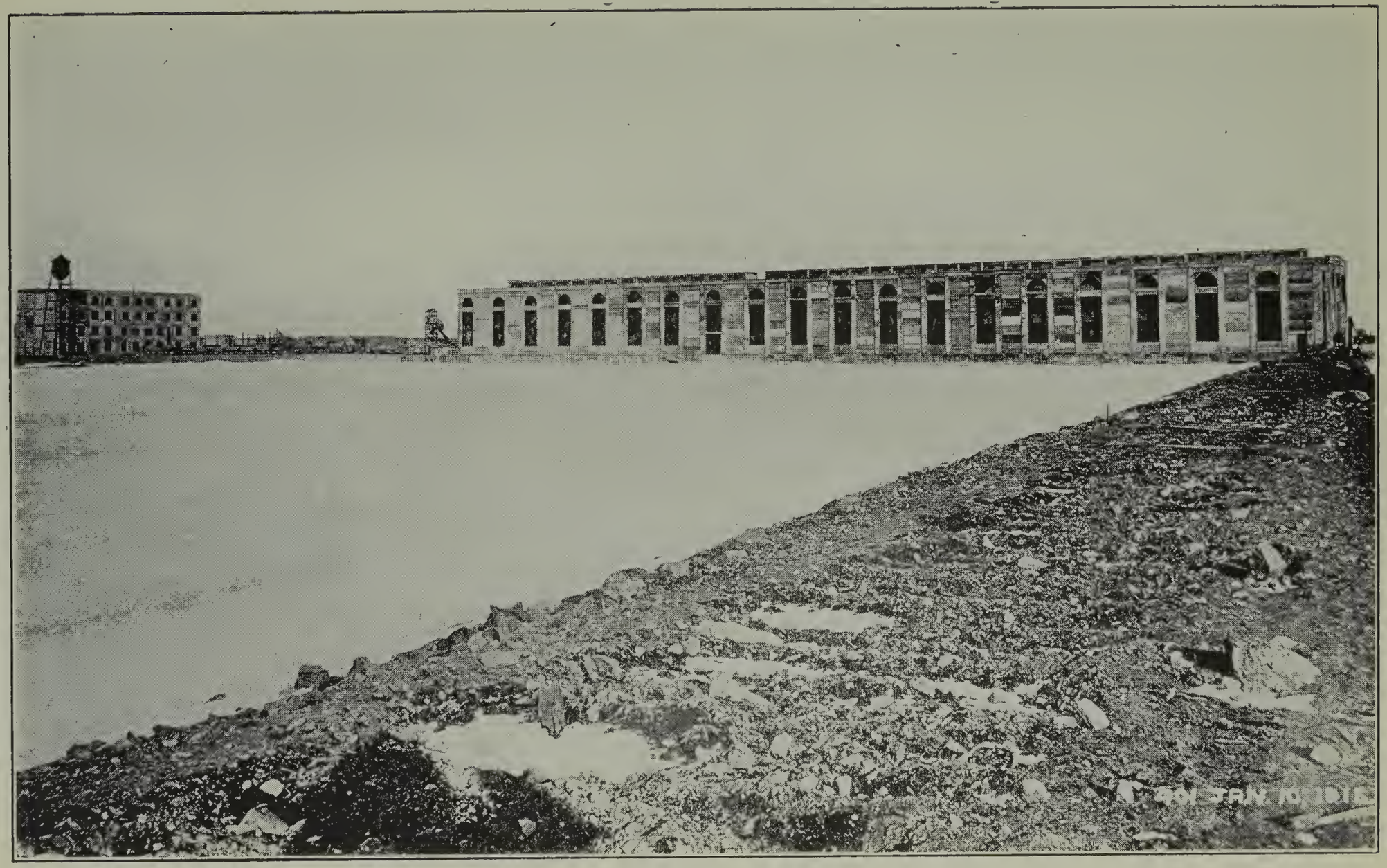


FIG. 9.

FIG. 10.

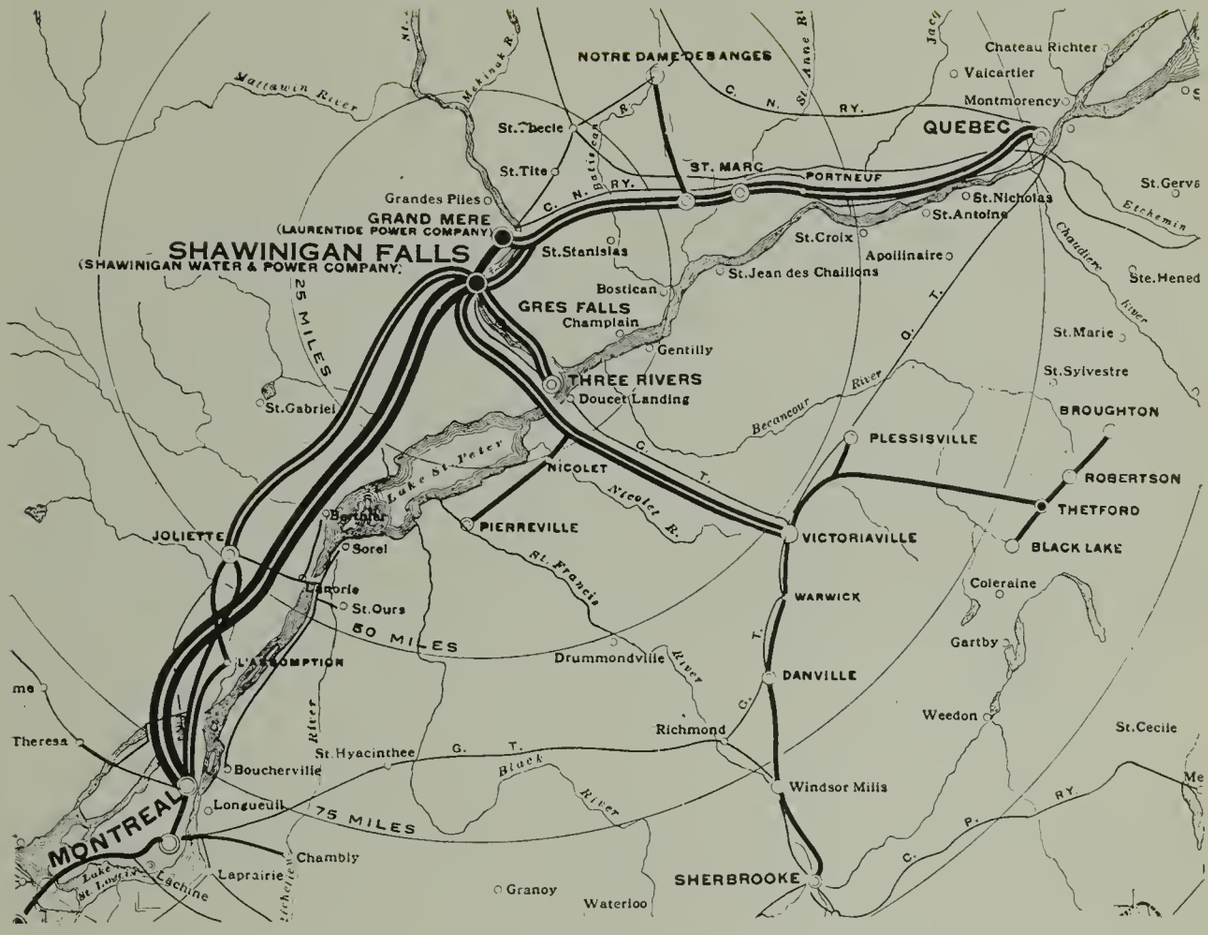

NOTRE DAME DES ANGES
C. N. RY.
St. Anne R.
Chateau Richter
Valcartier
Montmorency
QUEBEC
Matawin River
Matawin R.
St. Becle
St. Tite
Batiscan R.
ST. MARC
PORTNEUF
C. N. RY.
Grandes Piles
GRAND MERE
(LAURENTIDE POWER COMPANY)
SHAWINIGAN FALLS
(SHAWINIGAN WATER & POWER COMPANY)
St. Stanislas
St. Croix
St. Nicholas
St. Antoine
St. Gervs
Etchemin
Chaudiere River
Ste. Hened
Apollinaire
St. Jean des Chaillons
Bostican
GRES FALLS
Champlain
Gentilly
25 MILES
St. Gabriel
THREE RIVERS
Doucet Landing
G. T.
St. Marie
St. Sylvestre
Becancour River
BROUGHTON
PLESSISVILLE
NICOLET
Lake St. Peter
Nicolet R.
ROBERTSON
THETFORD
BLACK LAKE
JOLIETTE
Berthier
Sorel
PIERREVILLE
St. Francis
VICTORIAVILLE
Coleraine
Laprorie
St. Ours
WARWICK
50 MILES
L'ASSOMPTION
Drummondville
River
Gartby
DANVILLE
Richmond
Weedon
St. Cecile
Theresa
St. Hyacinthe
G. T.
Boucherville
Black River
Windsor Mills
MONTREAL
Longueuil
75 MILES
Chambly
Lake St. Louis
Lachine
Laprairie
SHERBROOKE
Granoy
Waterloo
C. P. RY.

FIG. 11.

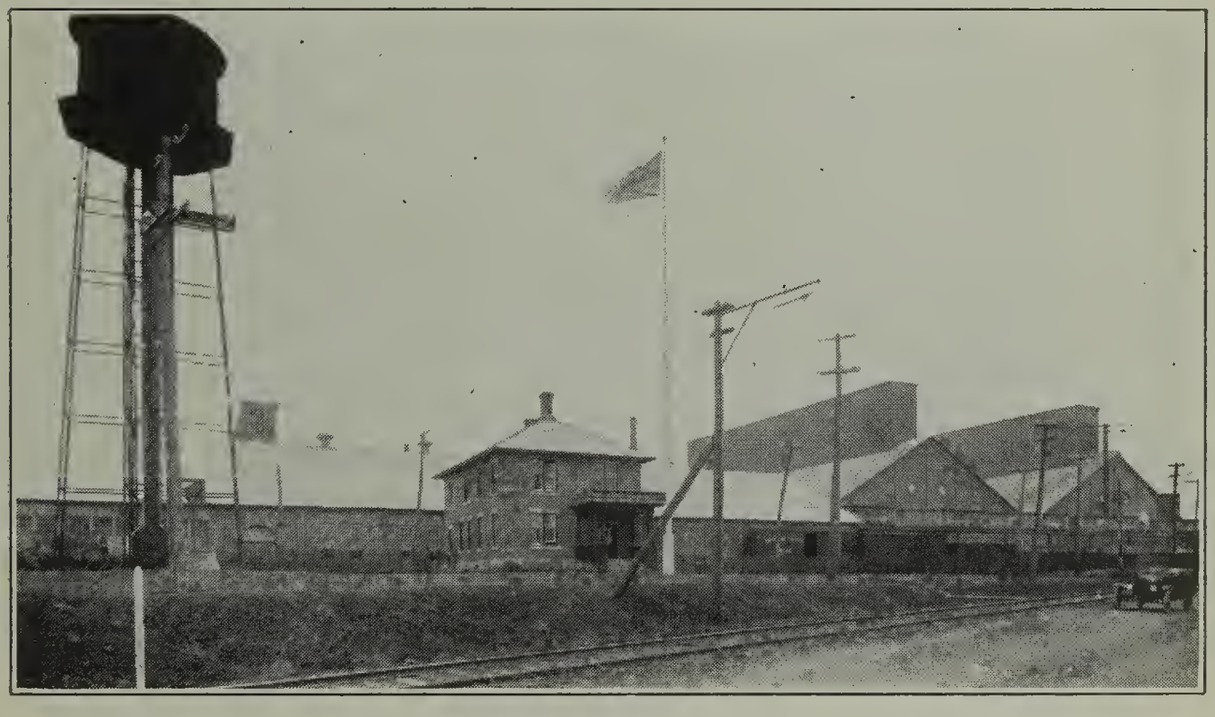


FIG. 12.

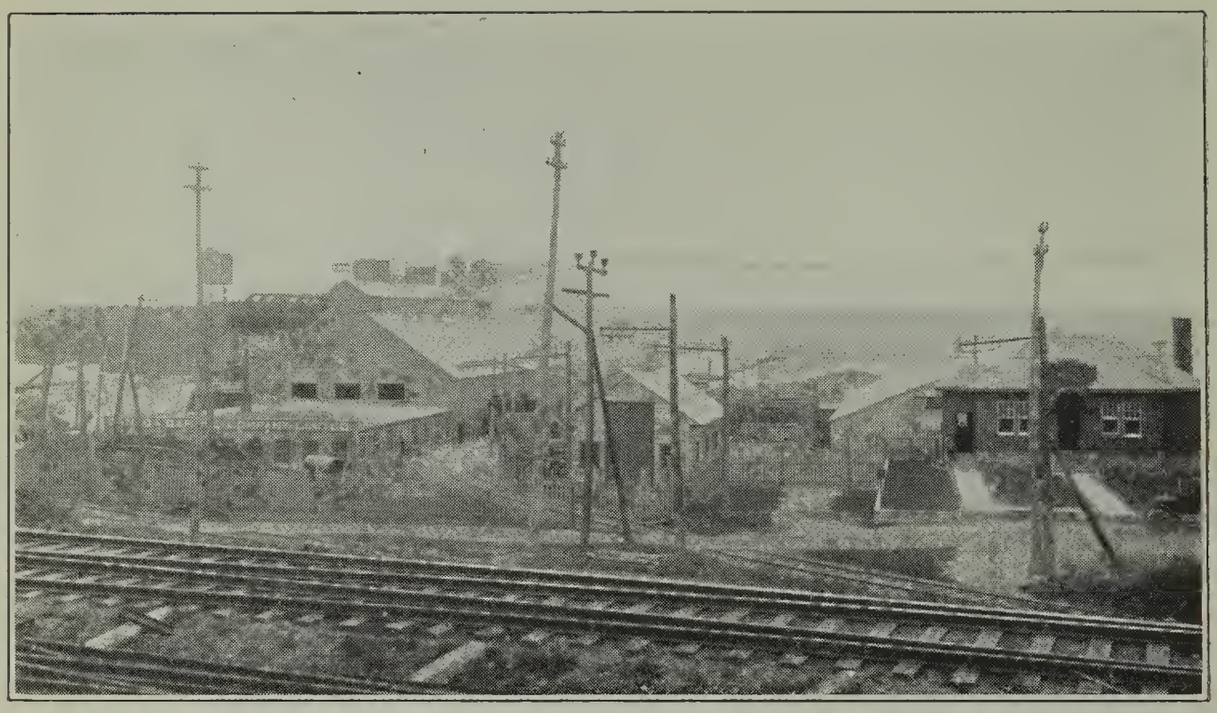


FIG. 13.

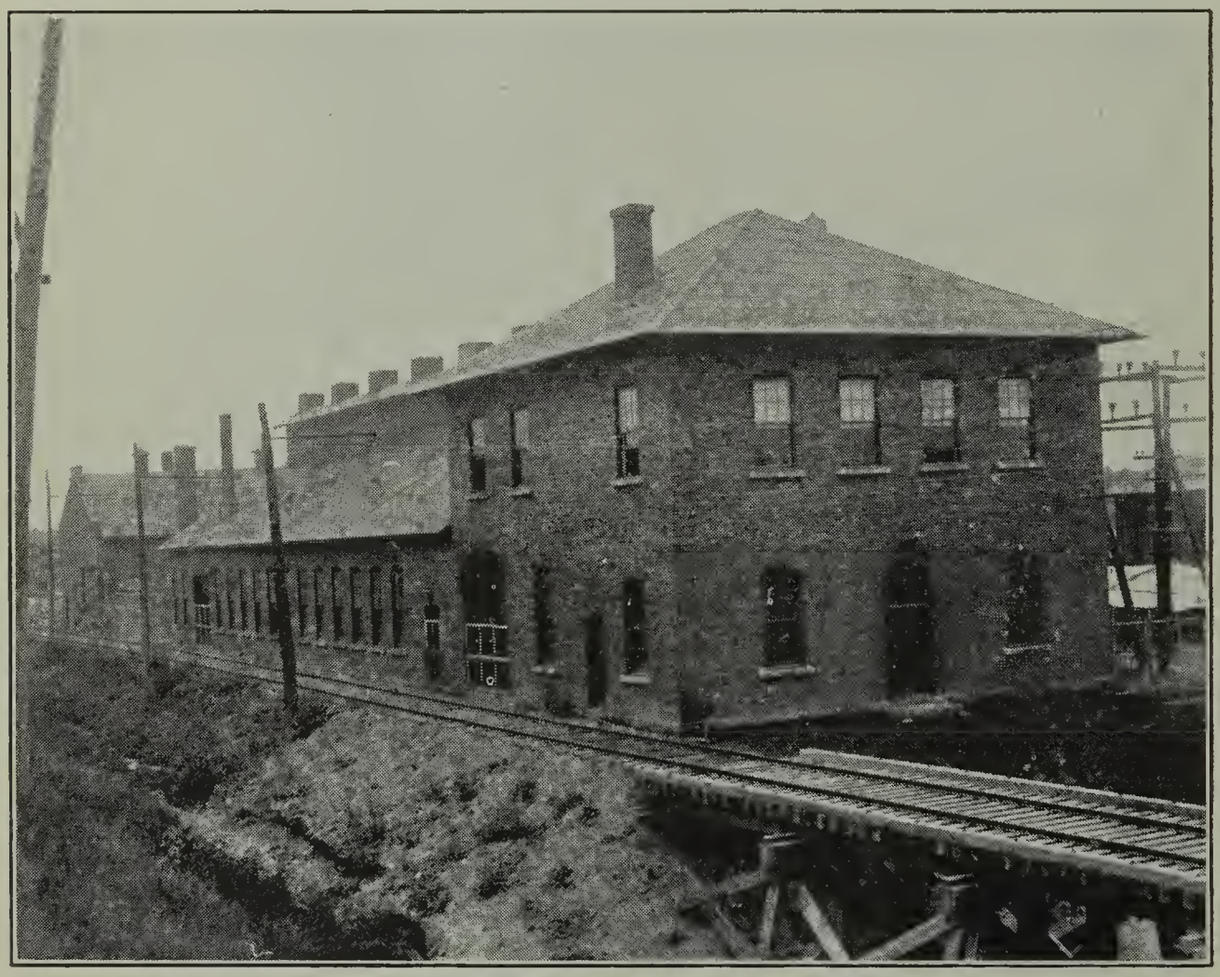


FIG. 14.

Fig. 15.

Fig. 16.

FIG. 17.

FIG. 18.

FIG. 19.

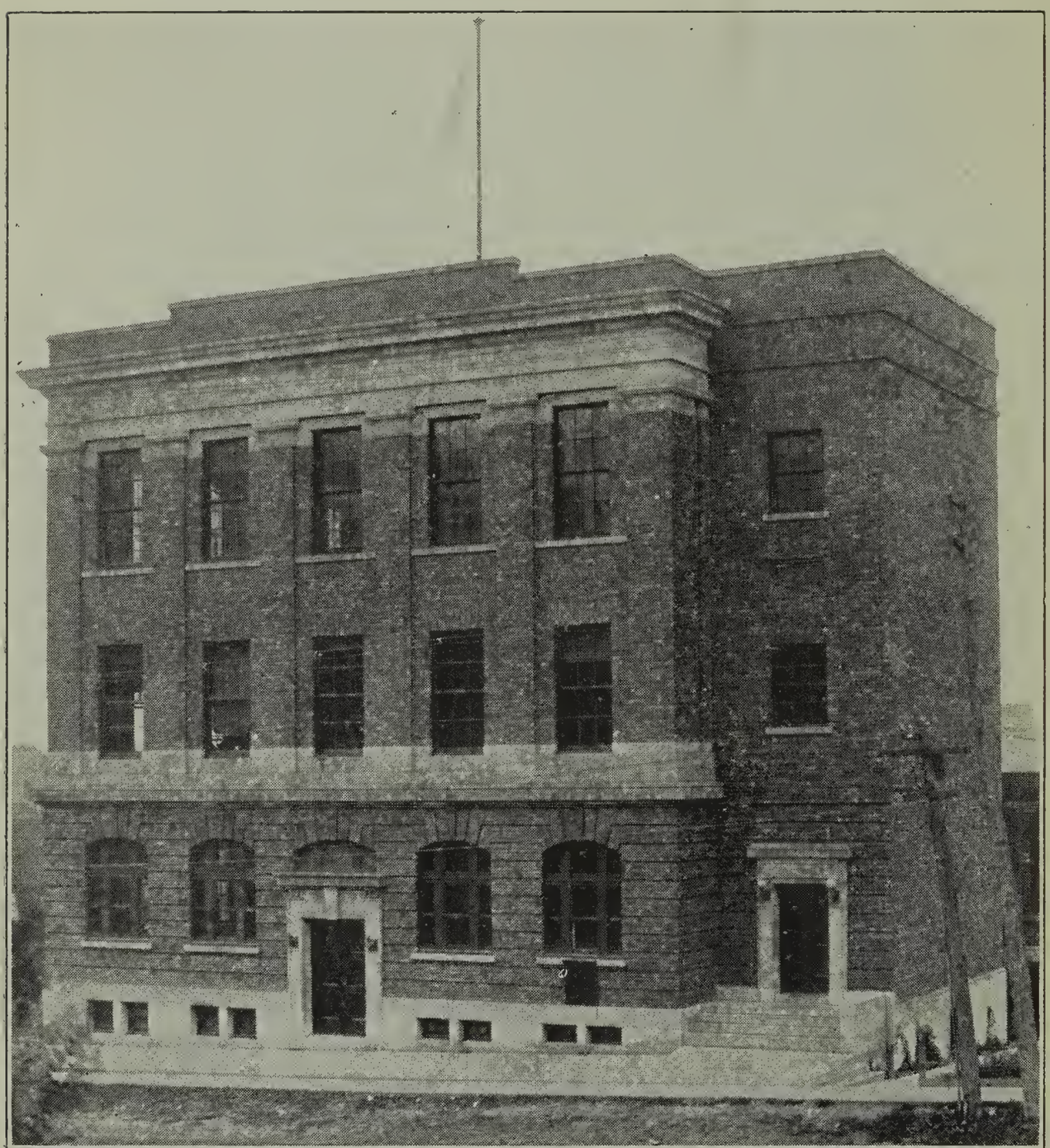


FIG. 20.

Shawinigan power is delivered in large quantities to the City of Montreal, and the picture of the Civic Investment Company's 120,000 H.P. plant at Cedars, Quebec (Plate 9), 30 miles from Montreal, is shown to indicate that a large amount of the Montreal load can be supplied from this plant in the future, thus leaving Shawinigan generating stations available for electrochemical industries. In this regard it may be noted that the Cedars Company has water rights amounting to nearly 100,000 H.P. more, for which machinery is not yet installed. In addition, the Montreal load is supplied by other hydraulic plants controlled by the Civic Investment Company and other companies, aggregating 100,000 H.P.

As to the future, the Shawinigan Company owns the water rights and has plans under way for the development of about 75,000 H. P. at the Gres Falls (Plate 10), four miles below Shawinigan on the St. Maurice River, where a head of 65 feet (20 m.) is available. In addition, within 100 miles of Shawinigan, there are other undeveloped water powers aggregating 300,000 H.P.

Totalizing the present developed and possible power mentioned above, the immediately visible supply of power available for feeding the Shawinigan System amounts to 1,091,000 H.P., of which 555,000 H.P. is yet unsold.

The amount of power which can be developed on the St. Maurice River is only limited by the amount of water and the falls available. Considerations of scenic beauty have not been permitted to enter into the power situation, and since the entire St. Maurice water shed lies within the Province of Quebec it is reasonable to believe that no interference or abrogation of rights need be looked for. The broad general policy of the Province with respect to water-power development has been one of the outstanding features in the progressiveness of the Province. This policy is well exemplified in the construction of the largest storage reservoir in the world, on the upper reaches of the St. Maurice River, by the Provincial Government. The water thus stored is leased over a long period of years for use at times of low water to the various companies using it for power or other purposes. This storage reservoir, with a dam costing $2,000,000 (Plate 2), forms a lake 360 square miles (1,000 sq. km.) in area, and impounds sufficient water to generate 1,000,000,000 K.W.H. in the

power plants of Shawinigan. It is interesting to note that if the level of this lake were raised 6 feet (1.8 m.), the water would commence to flow into the Hudson Bay, 900 miles away, rather than into the St. Lawrence River (Plate 3).

Summarizing, it appears that there is now installed 511,000 H.P. of generating machinery feeding the Shawinigan system, and that there is now over 40,000 H.P. not used and available for immediate delivery, and an additional 60,000 H.P. will be available as rapidly as required. There is also 580,000 H.P. as yet undeveloped, and this with the 100,000 H.P. as yet unsold, makes 680,000 H.P. which is available for electrochemical industries in the St. Maurice Valley. This amount is almost wholly available for this purpose, because the growth of the heavy city loads outside the St. Maurice Valley will be amply taken care of by other power plants near the cities.

PRESENT ELECTROCHEMICAL INDUSTRIES.

The Pittsburgh Reduction Co., now the Northern Aluminum Co., was one of the first industries to locate at Shawinigan. Their works are located near the Power House, and are the only works not in the electrochemical district referred to later. American refined bauxite is brought in by rail and reduced to aluminum, between 50,000 and 60,000 H.P. being used for this purpose. The Aluminum Co. also has a wire-drawing and cabling plant, and manufacture all the aluminum wire made in Canada. The works, which are quite extensive and employ some 500 men, are shown on Plate 12.

All other industries lie in the electrochemical district about two miles up the river from the power houses, and receive power at 25,000 to 50,000 volts.

The Canada Carbide Co. (Plate 13) occupies some 15 acres of land conveniently located between the Canadian Northern Railway and the Canadian Pacific Railway, with sidings to each. The company employs some 350 men, manufactures calcium carbide and acetylene gas only, and utilizes some 40,000 H.P.

The Shawinigan Electro-Metals Co. (Plate 14) produces metallic magnesium in many forms, and occupies about five acres, with their various processes. They utilize some 2,500 H.P. in both alternating- and direct-current furnaces.

The Canadian Electrode Co. (Plate 15) manufactures the larger sizes of carbon electrodes, and has an output of about 15 tons per day. The majority of the output is used in Shawinigan, although a small tonnage is exported.

The Canadian Electro-Products Co. (Plate 16) manufactures acetic acid and acetone from acetylene gas supplied by the Canada Carbide Co. This plant has been put into operation since the war, and has become one of the large industries at Shawinigan.

The Prest-O-Lite Company (Plate 17) operates a compressing plant for acetylene gas which they purchase from the Canada Carbide Co. and which is distributed as dissolved acetylene by them.

Fraser, Brace & Co. (Plate 18) have an experimental electric furnace plant for the manufacture of various kinds of steels and for general experimental purposes.

The latest addition to Shawinigan power users is the Canadian Aloxite Company, a subsidiary of the Carborundum Company, which will utilize over 20,000 H.P. and is building an extensive plant covering some 15 acres near the upper end of the electrochemical district (Plate 19).

While not electrochemical, two other industries in the Shawinigan district are worthy of mention on account of the large amount of power they consume. The Laurentide Co. at Grand Mere, utilizing 28,000 H.P. for the manufacture of paper and pulp, and the Belgo-Canadian Co. at Shawinigan, utilizing 18,000 H.P. for the same purpose (Plate 19).

RAW MATERIAL SUPPLIES.

While electrochemical industries are as yet perhaps in their infancy, the vast amount of power which is being made available throughout the world will bring forth new processes requiring new raw materials. We are nevertheless today dealing with processes now in use, and an attempt is here made to indicate the sources of certain raw materials which are today largely used in electrochemical processes.

Coke can be obtained from the gas company in Montreal, made from West Virginia coal, and is therefore very low in sulphur and phosphorus. About 17,000 tons a month are made, and a large portion of this is now being used for house-heating pur-

poses in Montreal, not over 50 percent being used for industrial plants.

Petroleum coke is made at Montreal by the Imperial Oil Co. to the amount of about 400 tons a month, about half being made from mid-continent crude and the balance from Mexican crude oil.

Tar is produced to the amount of about 3,000,000 gallons (11,300,000 liters) a year by the Montreal gas company, a refining plant of some magnitude is in operation, and it is reasonable to suppose that in the near future successful methods of coking this tar will be developed. At present about 15,000 tons of pitch per year are available from the refining plant.

Limestone exists throughout the Province of Quebec along the St. Lawrence River, and may be obtained within reasonable distances of Shawinigan of an analysis suitable for most electrochemical processes.

Silica occurs in large quantities throughout the Province, and may be obtained within 100 miles of Shawinigan with not more than 1 percent lime. Silica analyzing 2 percent lime can be very readily and cheaply obtained.

Zinc occurs as a deposit of low-grade complex ore, running about 12 percent zinc, at Notre Dame, about 25 miles from Shawinigan. The mines are now being worked by two operators, with modern mills, and about 30 tons of concentrates per day is produced.

Chromium and titanium ores are found in large deposits in the Province, the former, however, being somewhat low in grade and the latter somewhat high in phosphorus.

Magnesite occurs in large quantities within 75 miles of Shawinigan, and can be obtained with an average analysis of not more than 5 percent lime.

Sodium chloride, bauxite, anthracite coal and some other raw materials must be brought in from outside the Province, but this same condition applies to most other electrochemical districts.

Bituminous coal is obtained either from the United States or from Sydney, Nova Scotia, and in normal times the greater supply of coal comes from the latter place, costing normally about $3.20 a ton on Three Rivers wharf.

The country to the west of Shawinigan being the foothills of the Laurentian Mountains, it is full of many kinds of raw mate-

rials interesting to the electrochemist, such as feldspar, phosphate rock, graphite, mica and probably a host of as yet undiscovered minerals.

In general, the Shawinigan district, with its nearness to tide water (20 miles—33 km.), probably is as well situated as any of the more recent electrochemical districts with respect to the supply of raw materials, and is especially well situated for the shipment of materials, as will be shown later.

SHIPPING FACILITIES.

Shawinigan is served by the Canadian Northern and Canadian Pacific Railways. All switching to the various works is done by the Shawinigan Falls Terminal Railway so that inter-company switching is conveniently and cheaply obtained, and the facilities of each railway equally placed before all industries.

Shawinigan is 20 miles (33 km.) from Three Rivers, on the St. Lawrence River, which has two miles of modern dock system capable of docking any boat navigating the river. The St. Maurice River between Shawinigan and Three Rivers is full of rapids and has never been canalized, so that no water transportation is available to Shawinigan itself. Under present conditions it is almost impossible to get export shipments taken at Three Rivers docks, except under certain conditions, but after the war, when increased shipping is available, Three Rivers will no doubt become one of the more important ports of Canada, as it is open nearly a month longer per year than Montreal.

Shawinigan is 90 miles from Montreal and Quebec, the Canadian summer ports, which are open and ice-free about seven months of each year. It is about 700 miles (1,100 km.) and 800 miles (1,300 km.) to St. John and Halifax respectively, which are the Canadian winter ports. The National Transcontinental Railway makes connection with the Canadian Pacific and Canadian Northern Railways at a point about 30 miles (50 km.) from Shawinigan and thus there are two independent railroads from Shawinigan to both the summer and the winter ports.

While it is difficult to discuss export shipping rates at the present time, it is nevertheless true that in normal times, averaging a large number of the ordinary heavier electrochemical products, a differential of $2.00 a ton exists in favor of Shawinigan in summer

and $1.00 per ton in winter over Niagara Falls, which city is taken as representing the greatest electrochemical center on the continent. These differentials will of course only apply to products which can be exported to ports reached by the Canadian sailings, which in general are satisfactory for supplying the European trade. If products are destined for ports not reached by Canadian sailings, it will in general be cheaper to use New York as a shipping point, in which case a differential of between $1.00 and $2.00 a ton exists in favor of Niagara Falls.

A feature which is of great help at the present time is the ability of Shawinigan exporters to ship on a through bill of lading from Shawinigan to any port reached by the Canadian Pacific Railway in Europe or Asia. This means that freight need only be delivered to the railway company, which will thereafter assume all responsibility and deliver the freight at the port desired. In normal times it is also possible to arrange through export shipments on the Canadian Northern Railway. Even if this advantage is not made use of, the excellent export facilities and harbors at Montreal, Quebec, St. John and Halifax, make exporting relatively easy, and it is said that the facilities of Montreal Harbor are not excelled anywhere on the Atlantic seaboard.

For exporting to the United States, an additional railroad, the Grand Trunk, is available at St. Gregoire, opposite Three Rivers, thus affording three competitive routes to American points.

It would be quite difficult to analyze the Shawinigan-United States shipping situation, as this is so largely a function of the product and the point to which it is shipped, but again discussing Niagara Falls and taking Pittsburgh as the destination, a differential of $3.40 and $2.40 per ton exists in favor of Niagara Falls for fourth- and fifth-class freight rates respectively.

If American raw materials are imported to Canada to be manufactured, and if an import duty is paid, a drawback of 99 percent of this duty can be obtained when the manufactured products are exported to the United States, so that the cost of working up imported raw materials is not increased appreciably by import duties.

In this shipping discussion the importation of raw materials has not been discussed, as they come from such widely separated points and in general are brought from those points from which the

lowest freight rates exist to the power development, and are so much a function of the particular electrochemical industry that no general analysis is possible.

For certain materials, which can be imported in ships, Shawinigan and Three Rivers (the latter being a tide-water port) would probably enjoy a lower freight rate than interior points, and the electrochemical development at Three Rivers will probably rest on this advantageous importation of raw materials by sea.

Summarizing, it would appear that for export to Europe, Shawinigan enjoys a distinct advantage over inland points, but that for shipments to Pittsburgh and American industrial centers about an equal disadvantage exists. It would therefore seem that Shawinigan would offer a reasonable location for electrochemical plants serving the European market.

MANUFACTURING CONDITIONS.

The St. Maurice Valley, from Grand Mere to the St. Lawrence, a distance of 30 miles (50 cm.), has a population of about 100,000 people; of these 8,000 live in Grand Mere, 12,000 in Shawinigan, and 25,000 in Three Rivers, leaving over 50,000 people in the farming districts surrounding these cities. The population is largely French, and as the families are large, a relatively large amount of local French labor is available. This labor is not migratory, and when under French foremen is of a satisfactory character. Labor troubles in the Shawinigan district are practically unknown, and not one of the Shawinigan industries has ever been shut down owing to labor disturbances.

The well-populated farming territory surrounding Shawinigan provides relatively cheap food stuffs, and this, combined with the natural French thrift, makes labor somewhat cheaper in this district than perhaps in any other electrochemical industrial location. It is notably true that at the present time of very high labor costs, common labor is obtained at Shawinigan for 25 cents an hour.

Shawinigan, like other large rapidly growing communities, is somewhat hampered at the moment by the lack of housing facilities, but houses are being rapidly built and will no doubt keep pace with the increasing population. As an indication of the stability of the labor, it should be noted that a great number of the houses in Shawinigan are owned by the workmen themselves.

There is a technical institute in the city (Plate 20) having some 150 students and equipped with fine modern laboratories housed in modern buildings. This institute offers a means of education to the younger men, in order that they may be more fitted for the industries existing in the community, and its night courses, which are well attended, aid workmen to become more useful and to become foremen and superintendents.

As the community is quite new, large amounts of land are available for industries, and in general sites may be found lying between the railroads, so that siding facilities may be had on each. There is no difficulty in obtaining water from the river for industrial purposes, and this water is of such character that it can be used by the majority of industrial works.

As the St. Maurice River is not used for drinking purposes below Shawinigan, and as the salt sea water is met a few miles below Three Rivers, very little if any objection can be made to stream pollution by industrial plants.

RESUME.

Summarizing the Shawinigan electrochemical situation, it would appear that the present electrochemical industries have

1. Cheap power.
2. Cheap labor.
3. Low import rates for ocean-borne raw materials.
4. Low export freight rates.
5. High coal costs.
6. High freight rates to American points.
7. Access to a large amount of power.

The number and size of the electrochemical industries at Shawinigan must increase rapidly in the future, because with 100,000 H. P. now available as fast as plants can be erected to utilize it, and with over 600,000 H. P. yet untouched, and with its low power costs, cheap labor, and advantageous export rates, it offers a strategic location for serving the European market, which for many years to come must be a large one. At the moment it is one of the few localities in the country where power in quantity can be obtained quickly and cheaply, and where the necessary

labor for plant operation can be obtained without excessive importation.

With these present advantages and future possibilities, it is almost certain that Shawinigan will continue to be the electrochemical center of the Dominion of Canada.

September 6, 1917.

DISCUSSION.

C. G. FINK[1]: I have been following this question of water power pretty closely for the last few years. It is a source not merely of satisfaction but of consolation, that our neighbor has 550,000 horsepower for immediate use, and our friends at Niagara Falls might be interested in moving there if our Government does not come forward promptly with assistance.

H. E. RANDALL (*Communicated*): Our President puts the matter a little too strongly when he states that 550,000 horsepower is available for immediate use. While this amount of power is available probably as fast as plants can be erected to utilize that amount of power, nevertheless the amount of power actually immediately available is somewhat over 40,000 horsepower, which can be increased to 100,000 horsepower within three months.

[1] Director of Research, Chile Exploration Co., New York City.

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THE SUBSTATION PROBLEM OF THE ELECTROCHEMICAL PLANT

By J. L. McK. Yardley.*

[Abstract.]

The author discusses the relations between the electrical apparatus conditions and the electrochemical conditions, that is, between the factors conditioning efficiency or inefficiency of the electrical installation and the factors imposed by the nature of the electrochemical cells and their arrangement into plant units. The discussion is illustrated by details of aluminum and copper plants and caustic soda installations. The voltage variation required is discussed, particularly in regard to the adaptability of special electrical plant to meet certain variations in cell room requirements. Photographs of seven typical electrical installations are shown, and briefly described.

No attempt will be made to consider this subject in all its aspects, and it is probable that none of the features mentioned will be treated fully. It is intended, rather, to give a sketch or outline of the more obvious factors bearing on the matter, and to discuss only some of the simple and fundamental particulars. It is desirable to review the subject in this way from time to time for the reason that many of the same points come up for consideration each time a new electrochemical plant is planned, and experience shows that things which are well known and which are fundamental and simple are continually being forgotten or overlooked.

The principal conditions which determine the substation layout and the nature of its equipment are:

(1) The nature and magnitude of the process; that is, the amount of current required per unit cell, tank or pot room, the

* General Engineer, Westinghouse Electric & Mfg. Company.

voltage range, the length of time; also the number of units and the ground space available.

(2) The nature of the power supply, particularly whether the voltage is high or moderate and whether it is practically constant or subject to variations.

(3) The inherent characteristics and costs of available apparatus.

Owing to the intense activity in all the electrochemical industries, such as zinc, copper, aluminum, chlorine and caustic soda, etc., during the past three years, a great variety of new propositions has come under consideration. In some of these at the outset the general arrangements have appeared to depend upon and have been determined by one or two of these sets of conditions rather than by all three. In the end it has usually been found necessary to give the same full consideration to each condition. It has been found impracticable to place all the emphasis upon the meeting of one set of conditions without giving due weight to the others. One could not consider or make a decision which was desirable from the process point of view to the exclusion or without adequate thought of the power supply or the available apparatus. In other words, the conditions imposed by the available apparatus and the power supply were found to be on the same basis as those imposed by the process, in determining the layout of the plant.

To most of us it doubtless appears perfectly obvious that this should be so. However, many individual mistakes, causing poor operating results and financial losses of a more or less permanent nature, have been due to a lack of appreciation of this point. There are a number of electrochemical installations in this country and elsewhere of which it is safe to say that better financial returns would be experienced had the same effort been made to adapt the process end (through change in size or arrangement of individual cell or tank room units) to the readily available apparatus as was made to obtain apparatus which would meet the special requirements of a predetermined process-end arrangement. Nevertheless, to a very great extent, there has been true co-ordination between the various parts in the majority of plants, and to a very great extent there has been success. It would be untrue to deny this.

Such mistakes and failures as have occurred have only tended to show that in the future there should be still greater co-operation between those whose efforts are able to bring about still greater success. Let us make as sure as we desire of the credentials of the man who wishes to co-operate with us, and then let us co-operate, without stint.

To take a very simple illustration of the sort of problem to be met, let us assume one desires to produce twenty thousand pounds (9,000 kg.) of caustic soda per day and has found that a total of approximately 252 electrolytic cells of a certain size are required. It is very doubtful that there are any good reasons from the process point of view why these cells should be arranged in three groups of approximately 84 in series in preference to four groups of approximately 63 in series; whereas, from the apparatus point of view there are good reasons in favor of the latter arrangement. Its requirements fall within the voltage range of apparatus which through years of use has become standardized the country over.

The manufacturers of electrical apparatus are wide awake to the rapid growth in the electrochemical industries, and they are keen to be leaders in that growth by adapting and applying their apparatus to its requirements in the most economical way, and by anticipating its future wants in new designs. However, it seems just as logical that the others who are interested in this development and want to make it an economical one, *viz.*, the inventors and manufacturers of cells, pots or furnaces and those who arrange them, assemble them, build them into a complete plant, and operate them—should work with equal or even greater zeal for a size and arrangement having requirements which, as nearly as possible, can be met by already standardized electrical apparatus. When one considers the relative costs of the electrical apparatus installation and the cell or furnace room installation, it is quite obvious that this should be so.

The manufacturers of electrical apparatus, supplying the electric lighting, electric railway and numerous manufacturing industries for thirty years or longer (a considerably greater period than there have been electrochemical industries of importance) had practically settled upon certain voltages and sizes which would meet the majority of ordinary requirements and insure economical manufacture. The incandescent lamp and the street railway motor

were responsible for the fact that much the larger part of the apparatus was built either for voltages in the neighborhood of 250 or for voltages between 500 and 600. These voltages were satisfactory to the various manufacturing industries as they came along, or else those industries turned to alternating current. Constructions for voltages near 250, or between 500 and 600, and in fairly regularly graded sizes, then became standardized. It was to the interest of the various industries that this should be so. The economy of manufacture became a maximum.

Thus we find the piece of electrical apparatus which, as much or more than any other, is used in the electrochemical industries, the rotary or synchronous converter, developed as it is today. The standard voltages are 250 and 600, and we find it developed in even sizes such as 100, 150, 200, 300, 500, 750, 1,000, 1,500, 2,000, 3,000, etc., kw. It usually happens, however, that, if the voltage requirement of the electrochemical process is somewhere within the approximate limits of 200 to 300 volts or 500 to 650 volts and the current requirement is equal to the kw. rating of the standard machine divided by its normal voltage, then the entire requirements may be met in a most economical manner by an already standardized piece of apparatus. This is particularly true for any process arrangement requiring a voltage between 200 and 300 and rotary converter units of 300 kw. each, or larger, since direct-connected alternating-current boosters have been developed for these machines so as to make it possible to readily vary the direct-current voltage delivered by the rotary converter 30 or 40 volts either side of whatever is the mean direct-current voltage obtained when operating upon a particular transformer tap.

Referring then, again, to the proposition to manufacture twenty thousand pounds (9,000 kg.) of caustic soda per day, if the cells are arranged in four groups of approximately 63 cells in each series since approximately four volts are required per cell, the total voltage required per group of cells will come within the scope of standard electrical apparatus; and, since the current capacity of the cell, in order to give this caustic soda output, must be approximately 1,200 A., the requirements of each group would be filled very exactly by one 300-kw., standard, 250-volt booster, rotary converter, with a 30-volt buck and boost range, and a step-down transformer giving a mean D. C. voltage on the rotary con-

verter of approximately 240. A total of four—or, in order to have a spare, five—300 kw. booster rotary converter transformer units would then be required.

The usual essential requirements of a unit cell or furnace are that it be efficient as to the use of both material and power, and that it be of simple construction, easy to handle and repair. To these we should add that either of itself, or in series-parallel combination with other duplicate cells, its power requirements should be such as can be supplied by standard electrical apparatus. One of the first and best chlorine cells took only 800 amperes, but a parallel arrangement of five circuits of the proper number of cells in series made a nice load for a standard 1,000-kw., 250-volt, booster rotary converter. It is interesting to note that some of the most recent chlorine cells have again only 1,000 to 1,500 amperes current capacity. In the interval, much larger cells have been built, cells of 2,000 to 2,300 amperes and even, according to reports, of 4,000 amperes capacity. Two circuits of 1,000 ampere cells make a nice load for a 500-kw. 250 volt booster rotary converter unit, and one circuit of 1,200 ampere cells, as above outlined, is a load for a 300-kw., 250-volt unit.

The aluminum industry very early appreciated the value of holding to recognized standards of voltage. The very great amount of power required to produce aluminum, approximately 15 kw.-hours for the reduction of one pound from the oxide, called for the best engineering efforts to obtain the most economical power-supply and substation and pot-room arrangements. In Europe, it became customary to employ in the reduction rooms a direct-current voltage of approximately 250. Since each pot required only about 6 volts, approximately 40 pots were placed in series, and since individual pots could readily be shunted and cut out of service for relining, no appreciable voltage variation on the pot lines was required. Rotary converter transformer units became the favored substation equipment, owing to their superior efficiency; and, while no variation of voltage was required at the pot-line terminals, it was thought best usually to provide the units with means for small voltage variations, to correct for variations in the power supply. In some cases this has been effected by employing rotary converters with direct-connected synchronous alternating current boosters. Generally, however, owing to the

anticipated variation in supply voltage being small, it has been effected by putting more than the normal amount of reactance in the step-down transformers and more than the normal amount of copper in the field coils of the rotary converters. Then the rotary converter voltage has been varied to meet line variations by varying the wattless component of the current input to the converter, which is simply done by varying the strength of the field current. A typical range for such an installation is 237½ to 262½ volts.

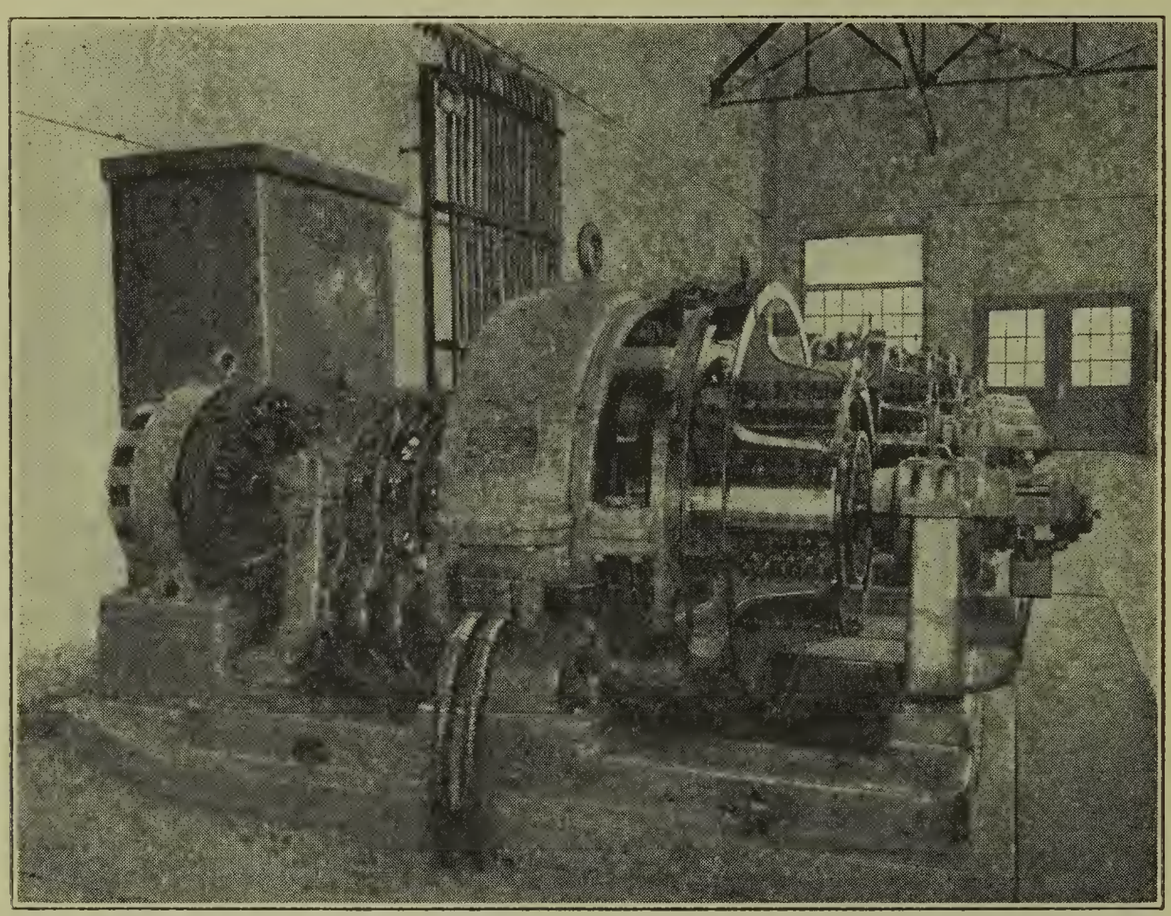


Fig. 1.

During the past two or three years, under the stress of war conditions, a good bit of work has been done which would probably have been done otherwise had it been thought there was time for thorough consideration and consultation with those who could help. The excuse for this sort of engineering in Europe has been that it was simply "provisoire." For instance, the electrical work in one large electrochemical substation of approximately 30,000 kw. capacity was done to a great extent with the aid of pencil sketches made at the time the apparatus was to be installed. The resulting blunders were to be compensated for by an earlier output.

Some of the history of this installation is rather interesting. The substation was for use in connection with a development of the Serpek process, in which, in addition to the fixation of nitrogen in the form of ammonia from aluminum nitride obtained from bauxite, a very pure alumina is obtained, to be afterwards reduced to aluminum in the electrolytic furnace. The first part of the process did not prove successful, but the production of aluminum went on, from alumina obtained elsewhere. It was planned to use about 5,000 kw. in a pot line; that is, 20,000 amperes at 250 volts, and the reduction plant was to consist of a number of pot lines. Power came from a 100,000 kw. hydro-electric generating station a short distance away, and the substation equipment was to consist of a large number of 2,000 kw. rotary converters provided with high reactance transformers and shunt field control of voltage, and a smaller number of approximately 1,000 kw. booster rotary converters. To supply each pot line it was necessary, therefore, that at least three rotary converters be operated in parallel on the direct-current side. The direct-current bus could then be sectionalized to this extent. However, owing to conditions which are practically bound to occur frequently in such installations, it was likely that all, or nearly all, of the total number of 14 rotary converters would at times be operated in parallel on the direct-current side.

In the original arrangement it was planned to connect these rotary converters to the direct-current bus by means of non-automatic switches. In the scheme as contemplated there was no automatic protection whatever on the direct-current side of the rotary converters, either against overload or reverse current. With this arrangement, then, in the case of a minor breakdown in either a rotary converter or its transformer, or the accidental opening of a field circuit or flashing over of a commutator, all the remaining machines would feed into the one experiencing the trouble, and before the alternating-current breakers could open would badly burn and damage it, at least, or would probably wreck it completely. Fortunately, there was an excellent construction engineer at hand who knew how rotary converters should be operated. When he learned of the proposed arrangement he made vigorous protest, and insisted that to protect against accident each rotary converter must be equipped on the direct-current side with

a carbon circuit breaker, supplied with an overload trip device and with a no-voltage release to be operated in connection with a reverse-current relay and the overspeed switch on the rotary converter. It is an interesting commentary on the way things of this sort sometimes work out, that while this engineer finally succeeded in having this auxiliary equipment added to the installation, it could only be done at the expense of making himself and his company so unpopular temporarily in his immediate surroundings that the order for the apparatus he recommended was placed with another manufacturer.

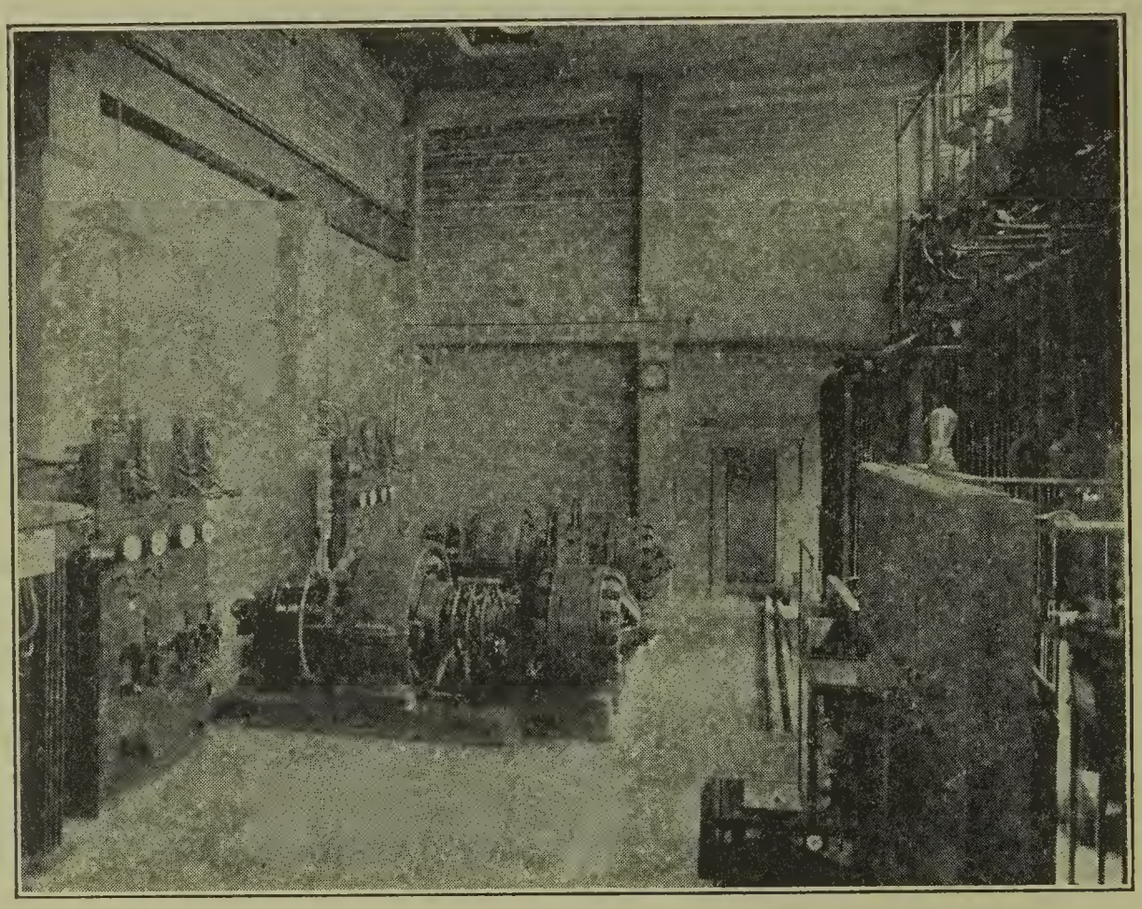


FIG. 2.

There was another bad feature in this installation which is worth describing for the reason that the same thing and variations on it are attempted repeatedly with varying degrees of success or failure. It was this: Four duplicate, commutating-pole, booster-type rotary converters, of approximately 1,000 kw. capacity each, were operated in parallel on the direct-current side to supply one line of pots, while on the alternating-current side two of them were connected to a common secondary winding of one step-down supply transformer, and the other two were connected to a com-

mon secondary winding of a duplicate transformer. The desire to operate two or more rotary converters from a common transformer is a natural enough one in many cases. The saving in cost of transformers per kw. is substantial. Occasionally, the saving in floor space of transformers is important. If it is desired to operate three or five or any odd number of rotary converters from the same transformer, this cannot usually be done with separate transformer secondaries, owing to difficulties in the transformer design. In the case of an even number, particularly in the case of two rotary converters, the transformer can almost always be very readily supplied with the same number of secondary windings and there is practically nothing saved in supplying it with only one secondary.

It is beyond the purpose of this paper to go in detail into the arguments for and against the operation of rotary converters in parallel on both alternating and direct-current sides. Such a method of operation is often proposed for electrochemical plants, and the arguments have been freely published. A brief history of the above particular case will, however, be instructive in showing the special consideration required to make the scheme operative. The rotary converters were to be started from the alternating-current end, by means of low-voltage starting taps on the transformer secondaries and double-throw knife-switches in the leads between the transformers and the rotary converters. When the first rotary converter was started, smoke began to roll off the second machine. It was then seen that it would be necessary to start both rotary converters from the common transformer secondary at the same time, an operation obviously entailing the simultaneous efforts of two men. In putting the four machines in parallel on the pot-room load, considerable difficulty was experienced at first, due to circulating currents causing very bad commutation and opening of the circuit breakers. If there had been a separate bus upon which the rotary converters could have been put in parallel and adjusted before being connected to the pot room load the matter would have been simplified. Finally, a scheme of operation was worked out by the construction engineer which it was necessary to follow exactly and which was as follows:

"(1) Start the two rotary converters connected electrically to one transformer at the same time on the starting tap. Then, open

the starting switches at the same time and throw both over to the running position. Repeat this operation with the two machines connected to the other transformer. Adjust for polarity on all four machines.

(2) Parallel one set of rotary converters through the direct-current breakers on the negative side only, within one volt; otherwise bad circulating currents will start to flow through the negative interpole windings with a certain amount of compounding, the circuit being completed through the alternating current ends. This

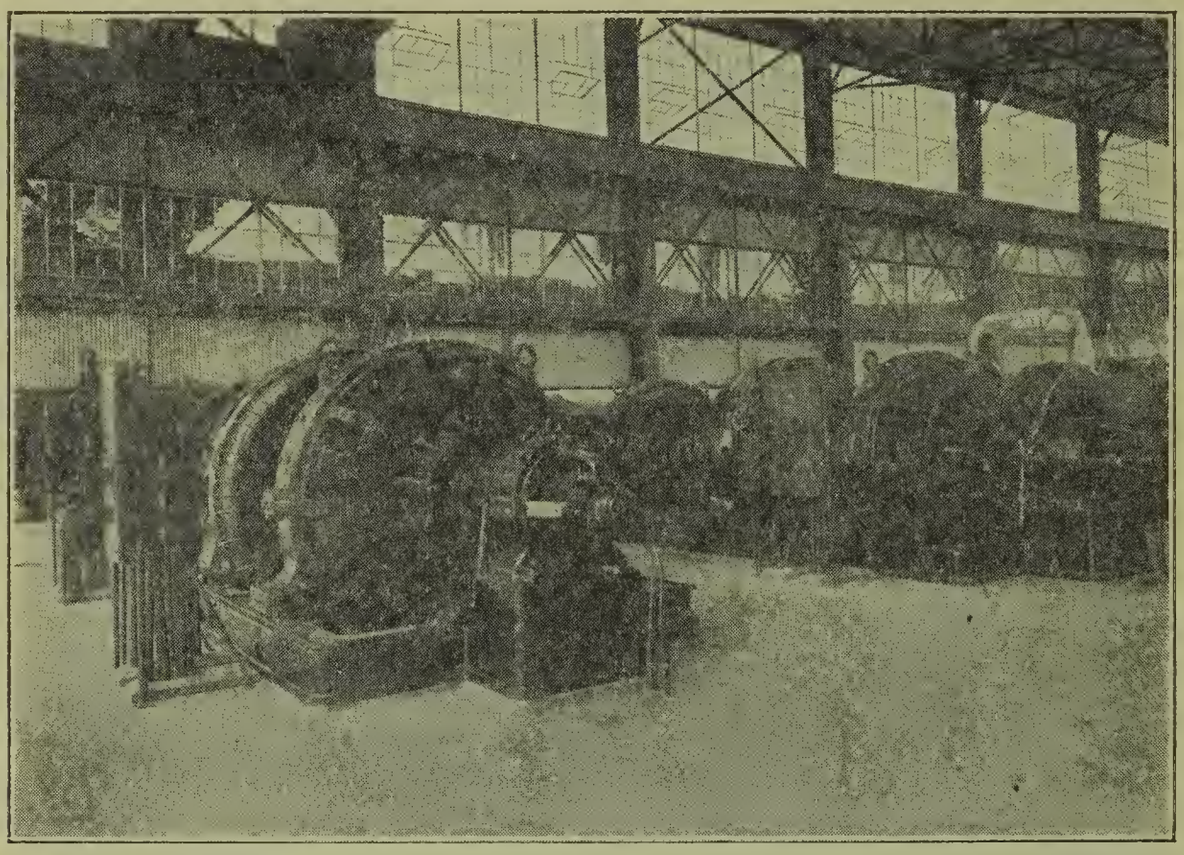


FIG. 3.

voltage adjustment is done with the main rotary shunt field rheostat, making sure that no direct current is circulating. Repeat the operation for the other two rotary converters.

(3) After the voltage on all rotary converters is set exactly, close the four switches (positive side of each machine) at the same time to the load in the reduction room. After these switches have been closed, quickly adjust the main shunt field rheostats for equal division of load; otherwise, circulating currents large enough to trip the direct-current breakers will start.

(4) No attempt should be made to employ the booster and commutating pole auxiliary field windings, as the circulating

current proposition cannot then be kept to a minimum. Operate the machines as straight rotary converters. All voltage variations for the reduction rooms should be done with the alternators at the power house, by phone."

The plant was operated for some months in this manner and finally the rotary converters were put on separate transformer secondaries. Almost identical trouble was experienced in a chlorine and caustic soda plant here in America within the past two years, due to the attempted operation of a number of booster rotary converters from a common transformer secondary. The final result was that the transformer was changed.

In America, the power supply, substation, and reduction-room arrangements in the aluminum industry have been developed to a high state of perfection. More intensive effort has been directed thereon than in probably any other similar industry. The advantage of a higher voltage on the pot lines was soon appreciated, and all recent developments have been based on employing a voltage in the neighborhood of 500 to 550 D. C. There is a substantial gain in efficiency and cost over 250 volt operation. The electrolyte being exceedingly non-conductive, except when molten at a very high temperature, and being, in fact, isolated in the reduction pots, there are practically no leakage losses. This voltage is, moreover, entirely safe for the men on the pot-room shifts, for the reason that there is no conducting electrolyte which is free and because nearly everything outside of the pots and the electrical connections thereto is an excellent insulator. While there will undoubtedly be great developments in this industry in the future, owing to the growing demand for aluminum, it is unlikely that there will be any appreciable change in the voltage employed in the reduction rooms. In fact, it is doubtful that it will be of appreciable advantage to use a voltage materially above the standard 600 volts in any electrochemical plant. The maximum efficiency of the rotary converter is practically reached at 600 volts. The cost of the apparatus begins to increase above that voltage and the operating troubles multiply so rapidly that it is unlikely the process end can show sufficient compensating advantages.

In the electrolytic deposition of some of the other metals, such as zinc, copper, etc., and in the refining of these metals, there are

other and more difficult conditions to be met. It is often a very hard matter, and sometimes quite an impossible one, to obtain the desired results without employing a great deal of rather special apparatus. In these developments particularly, then, the electrical manufacturers can, if allowed, be of very great assistance in the working out of the general arrangement which will be the most satisfactory from the ultimate point of view.

The particular, and the immediately apparent, point is the great range of voltage required on the cell or tank room. Thus, apparatus having a great voltage range is required, on account of the

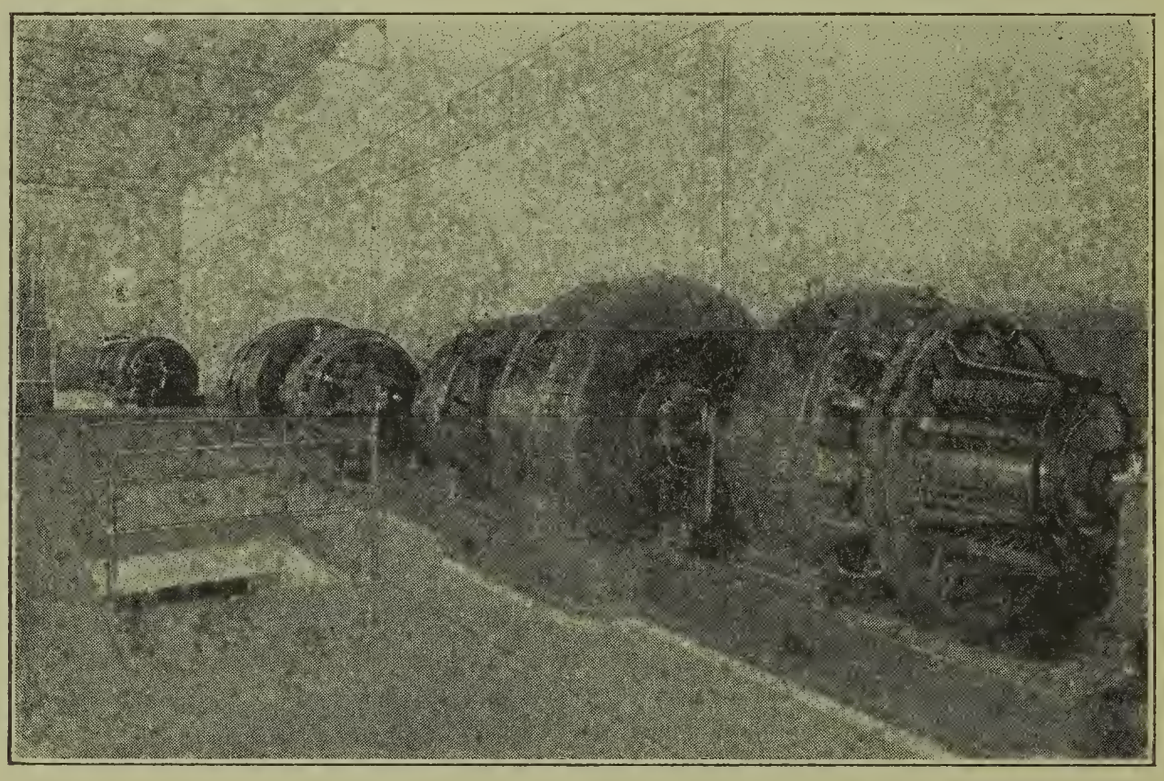


FIG. 4.

process and not on account of line variations; although, if there are line variations, the apparatus must be able to take care of them also. In this work a voltage range of 2 to 1 is quite a common thing and in some cases a voltage range of nearly 3 to 1 has been required. The reason for this great range appears to lie in the desire to be able to withdraw from the circuit large sections of the tanks for the removal of the metal and the treatment of the electrolyte. Where the liquor contains the metal, as by a process of leaching from the ore, it seems apparent that unless a very free circulation, affording practically continuous renewal, is achieved, there must be frequent withdrawal of what should be

large sections of the tanks. Since the deposition voltage of zinc is roughly twice that of copper, it is reasonable to suppose that a somewhat greater voltage variation might be desirable in a zinc reduction plant than in a copper reduction plant. In one of the latest installations for copper refining, the voltage range of the apparatus supplied was practically 2 to 1. Although the voltage for refining from the soluble copper anodes is only one-sixth, roughly, of the copper deposition voltage with insoluble anodes, it is necessary to carry in the neighborhood of 2 percent of insoluble-anode tanks, to correct for the gradual accumulation of copper in the electrolyte. These occasion an appreciable variation in the total resistance of the tank house when they are cut in or out of circuit.

Since, in a process of this sort, the electrolyte must be continually circulated to avoid stratification, as well as to permit purification, and since the electrolyte is highly conducting, the opportunities for current leakages are large. These leakage losses may be reduced to a minimum by careful planning of the tank-room arrangement, but they cannot be eliminated. The higher leakages in the higher voltage installations tend to offset the other advantages of such higher voltage. In fact, one refining company which had been operating a large part of its tank room with the electrodes in a series system and had previously purchased a large number of booster rotary converters having a normal voltage of 225, recently installed a new plant to operate on the multiple system and purchased for it a large number of booster rotary converters having a normal voltage of 110, although more than 3 percent in the rotary converter efficiency was lost thereby.

It is usually found that a process requiring this great voltage range can be supplied more satisfactorily by an installation of booster rotary converters than by any other type of installation. I do not mean to say that the booster, itself, gives the rotary converter this range. It is obtained by supplying one or two taps on the primary or secondary of the transformer and by providing the necessary switching for quickly transferring the booster rotary converter from one tap to another. In case the process should demand that the voltage be varied from one extreme of the range to the other, without even a momentary interruption of the load, a 3 to 1 range could not be met and a 2 to 1 range could scarcely

be met efficiently and economically by a booster rotary converter. There apparently are few or no cases where an absolutely continuous range is essential, but there have been cases in which the cost of the additional switching and the connections to the extra transformer taps required by the booster rotary converter installation has assisted in making an installation of motor generator sets appear favorable in comparison.

Motor generator sets have been applied in this work to a considerable extent, even within the past two or three years. When

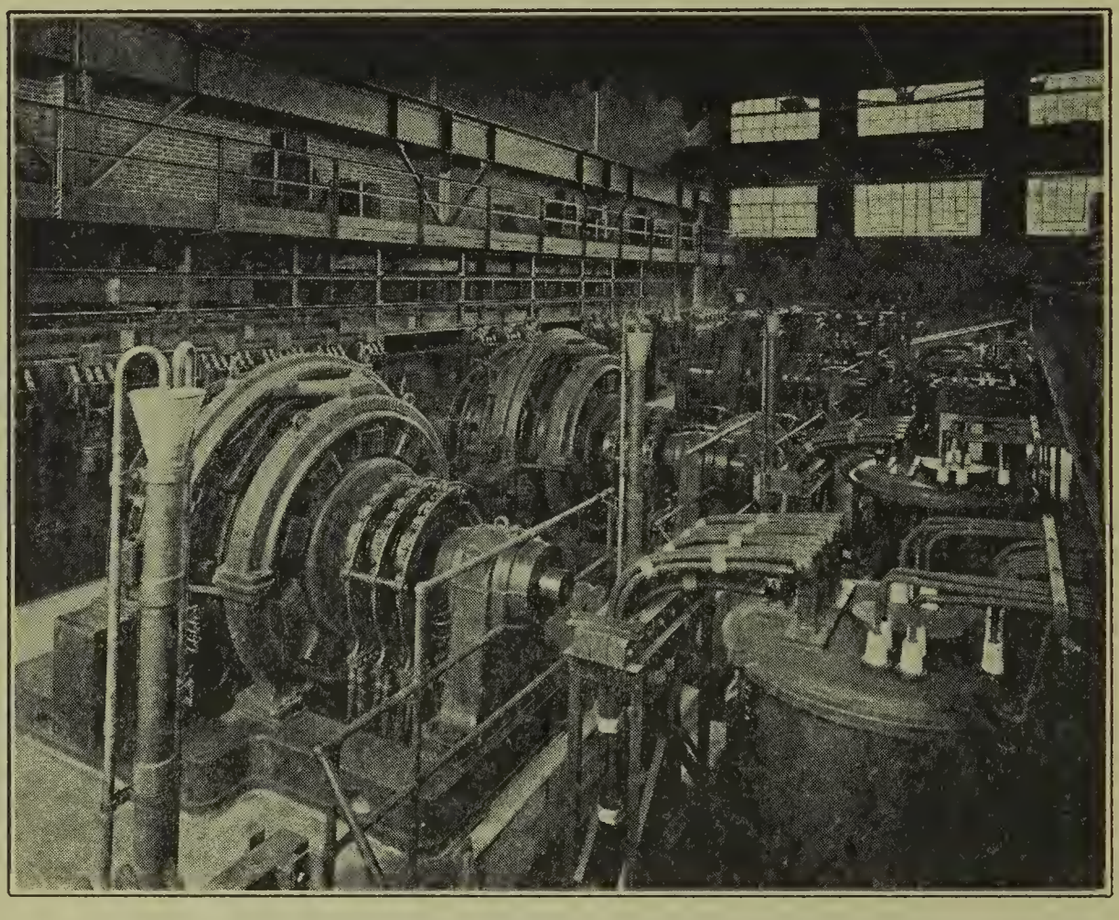


Fig. 5.

the voltage range is very large, a separate exciter is required; but as mentioned, the range is continuous. They also contain the possibility of power factor correction, which is sometimes availed of. When they appear to be an attractive commercial proposition, however, it is, I think, I may say, always the case that there is plenty of power available at a moderate voltage, 13,000 volts or less, from which to operate the motor generator sets without the added cost and losses of step-down transformers. Compared with this arrangement, there is a difference in efficiency of only in the

neighborhood of 2 percent in favor of a booster rotary converter transformer installation, assuming fairly continuous operation at the rated load; whereas, if transformers are required for the motor generator sets, due to the supply voltage being materially above 13,000 volts, there is a difference in efficiency in favor of a booster rotary converter installation in the neighborhood of 5 percent. It is this matter of efficiency, taken in conjunction with the cost of power, which is usually the determining factor.

The greatest voltage range, so far as I know, for which booster rotary converters have been provided is in the order of 2.5 to 1. This range is obtained in this case by means of one tap on the secondary winding of the transformer. It is usual in this relatively low-voltage work for the transformer to have a very small number of turns in its secondary winding, and in this case there are only three turns. A tap is brought out at the second turn, which is used for operation on the lower part of the range. This same tap is used in the starting of the rotary converters, although at the time of starting a machine, to prevent an excessive starting current, a suitable reactance is placed in the circuit, which reactance is short circuited by a switch when the rotary converter is in synchronism.

It often happens that the number of secondary turns requisite for an efficient and economical design of transformer will not permit exactly the desired tap being obtained on the secondary side. On the other hand, if the supply voltage is high, such as 66,000 volts or 110,000 volts, it is obviously impracticable to place the tap on the primary winding, on account of the expensive switching. In such case it is evident that some change must be made in the number of cell or tank sections, in order to change the range one way or other. Sometimes the line voltage is stepped down in two transformations and the voltage on the primary of the rotary transformers is 13,000 volts or less. In such cases, if the transformer secondary current is very large, so that secondary switches would be expensive and difficult to handle, the primary tap and primary switching provide a very satisfactory arrangement.

If the voltage at the primary of the rotary transformer is 2,200 volts, 6,600 volts or even 13,000 volts, there is an arrangement of

apparatus which, assuming the conditions of the process are just suitable, may prove economical. The arrangement which has been suggested in a number of cases is to employ straight rotary converters with separately-driven boosters, instead of booster rotary converters. Assuming that there are long periods of operation in the process at the lower extreme of the voltage range, or even at the middle of the voltage range, then it is reasonable to expect a saving in power by the use of separately driven boosters which can be shut down when not required. Considering a unit of large

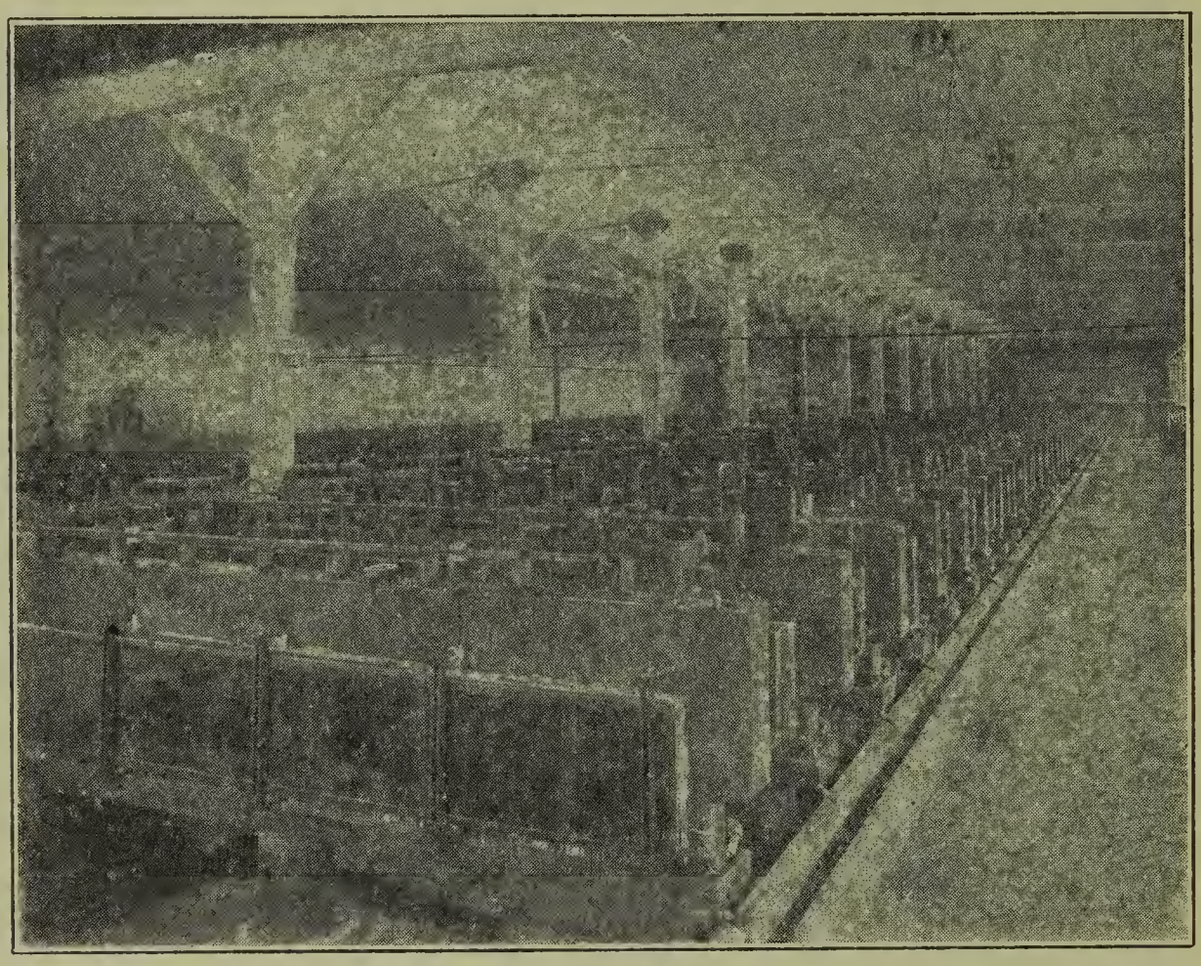


FIG. 6.

size, the combination of a straight rotary converter and a separately-driven booster, operating at the extreme of the voltage range, that is, with the booster operating at full load, has an efficiency which is practically equal to, or may even slightly exceed, that of the corresponding booster rotary converter. However, when operating at other points of the range, the efficiency is in favor of the booster rotary converter; and, when operating at or near the no-load condition of the booster, which may be the lower extreme of the voltage range of the process, or the middle of the

range, depending upon the size of the booster, there is a considerable difference in efficiency in favor of the booster rotary converter.

It has not seemed likely that many processes would permit of operation for long intervals at a voltage corresponding to the no-load condition on the booster, so that the shutting down of the separately-driven booster could be definitely taken into the calculation. However, unless the process conditions do permit of such operation, there do not seem to be many good reasons for employing this combination. It is true, the separately-driven booster makes the commutating conditions easier in the straight rotary converter than in the booster rotary converter, but this is not a sufficient argument. So far as I have been able to find by investigation, there is nothing saved in first cost when one separate booster is supplied for each rotary converter, as compared with supplying the same number of units of booster rotary converters. I made some investigations in connection with a case in which it was proposed to use a total of 40,000 amperes direct current over a range of 580 to 510 volts, and was required that the 40,000 amperes should be used in four 10,000 ampere units. Assuming a process with a short "starting period" at 580 volts and a long running period at 510 volts, it seemed possible that something might be saved in first cost by supplying separately-driven boosters, only two of them, for four straight rotary converters. It became apparent, shortly, that this could not be done if the boosters were placed in the circuit between the transformers and the rotary converters. This was on account of the great expense of the transfer bus and switches required by the arrangement. It was found, then, that if a moderate voltage on the primary of the rotary transformers, such as 6,600 volts, could be assumed, and if the two separately-driven boosters and the necessary transfer bus and switches were placed in that circuit, there would be a slight saving in first cost over an installation of four booster rotary converter units. The saving appeared to me to be in no way commensurate with the great convenience of having a booster for each unit and of having that booster self-contained in a booster rotary converter. To show how completely the power saving is determined by the cycle of operation, the following figures are given:

With cycles of operation consisting of the following:

(1) One hour at 580 volts and 23 hours at 510 volts, with the separately-driven boosters shut down, there are 1,450 kw. hours of losses showing against the booster rotary converters.

(2) Six hours at 580 volts and 18 hours at 510 volts, with the separately-driven boosters shut down, there are 1,300 kw. hours of losses showing against the booster rotary converters.

(3) Six hours at 580 volts, 6 hours at 545 volts, 6 hours just above 510 volts, and 6 hours at 510 volts, with the separately-

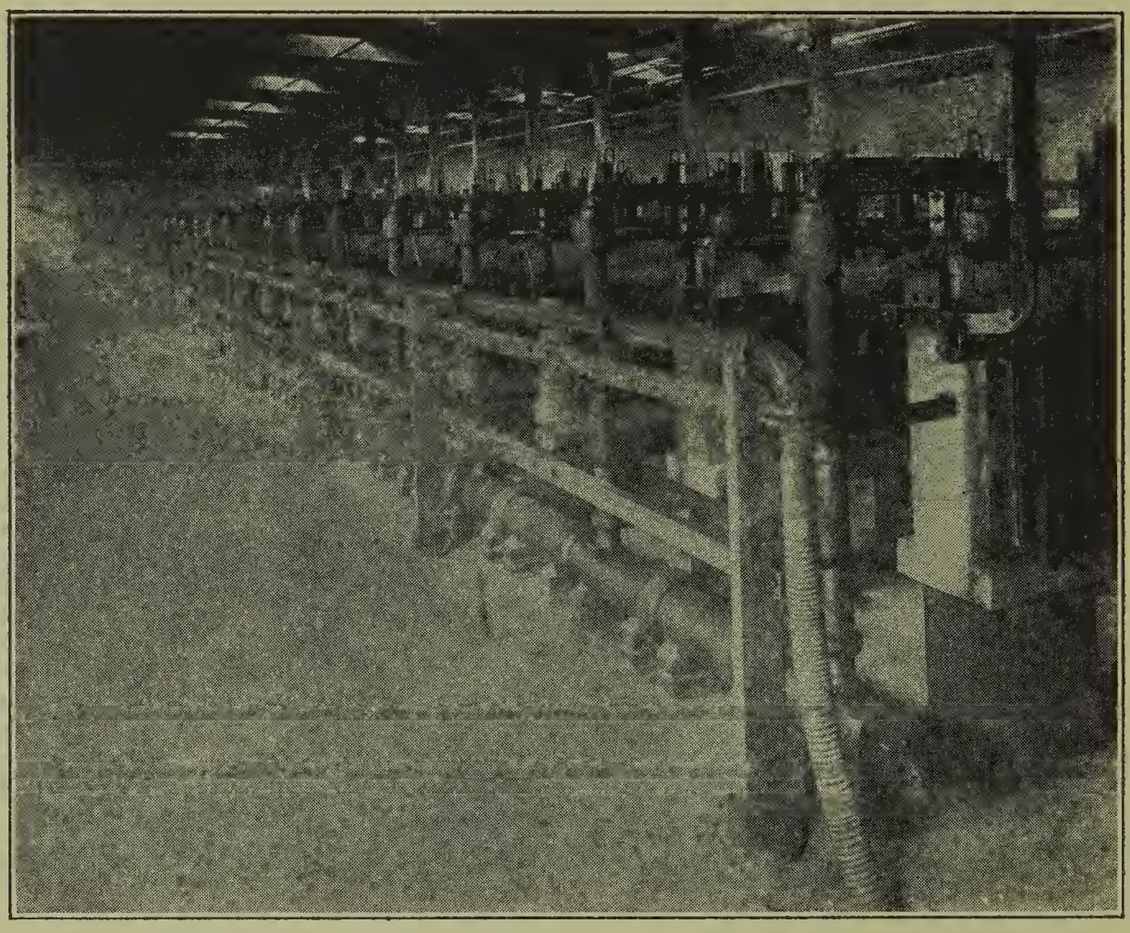


FIG. 7.

driven boosters shut down, the losses are practically the same in each arrangement.

(4) Six hours at 580 volts, 6 hours at 545 volts and 12 hours at 510 volts, or just above 510 volts, with the separately-driven boosters running continuously, there are 1,000 kw. hours of losses showing against the combination of straight rotary converters and separately-driven boosters.

Assuming a cost of power of about 0.3 cent per kw. hour, the above kw. hour losses may be read as dollars per year.

While it has been the intention to discuss the subject of this paper only in a very general way, hoping at the same time to refresh our memories on a number of points which are of special interest, it is felt that, from a general interest standpoint, the intention will have failed if all description of existing installations is omitted. Accordingly, a number of pnotographs are shown and a brief description of each is added:

Photograph 1 shows an installation of five 500 kw., 250-volt, 60-cycle booster rotary converters, supplying a voltage range of from 220 to 280 volts. The alternating-current leads are shown coming through the wall from the transformer beyond. This apparatus is used for the production of chlorine and caustic soda.

Photograph 2 shows part of the substation equipment of an electrochemical plant which makes chlorine and caustic soda. The machines shown are two 500 kw., 250-volt, 25-cycle, 220 to 280-volt standard-range booster rotary converters which are operated from a transformer tap to give a normal of 225 volts and a range of 200 to 250 volts. The photograph also shows the incoming 11,000 volt line and bus structure, the step-down transformers, the alternating-current switchboard and meter panels, the starting panels and the direct-current switchboard panels. This photograph is interesting, particularly for the reason that it shows practically the amount of apparatus which has been provided in several cases since the beginning of the war in small chlorine plants to provide bleach for pulp and paper mills, etc. In continuous operation, this amount of apparatus should produce roughly 7 tons of chlorine per day.

Photograph 3 shows part of an installation of motor generator sets in an electrolytic copper plant. There are three 4-unit motor-generator sets, each consisting of:

One 2,500-H. P., 1,930-K. V. A., 2,200-volt, 60-cycle, 100 per-cent P. F., 3-phase, 600 R. P. M., motor.

Two 850-kw., 170-volt, 5,000-ampere generators, having a range of from 90 to 185 volts.

One direct-connected 22½-kw. 125-volt exciter.

The direct-current breaker and switch panels for each generator are located at its side. The usual operation is at from 100 to 115 volts. This amount of apparatus in continuous operation should

be able to extract from solution roughly 40 tons of copper per day, or to refine roughly 240 tons of copper per day.

Photograph 4 shows part of a substation installation in an electrolytic refining plant. It shows three motor generator sets, each consisting of the following:

One 1,730-H. P., 6,600-volt, 3-phase, 60-cycle, 90 percent P. F., motor.

Two 600-kw., 200-volt generators, having a range of from 140 to 210 volts. Each generator delivers 3,500 amp. at 140 volts and 3,000 amp. over the range 180 to 210 volts. The operation is at 200 volts, 3,000 amperes the greater part of the time.

Photograph 5 shows part of the interior of a substation in a copper refinery. In this part there are four booster rotary converter units with step-down transformers and switching. The rotary converters have a total range of from 62 to 126 volts and have an output of 8,670 amperes each over this entire range. This range is obtained by means of one extra tap on the primary of the 13,000 volt transformer. There is no secondary switching. The 13,000 volt switches, located in the bus structure at the extreme right, are remote controlled from the alternating-current switchboard panels in the extreme rear. The rotary converters are started by throwing them directly on to the lower voltage tap. The main direct-current switch panels are shown at the left. This apparatus in continuous operation should be able to refine roughly 275 tons of copper per day.

Photographs 6 and 7 show typical cell rooms for the production of caustic soda, bleach and other chlorine products. Six is a front view, and 7 is a rear view which shows the electrical connections, brine mains, and chlorine gas mains. These cuts are of particular interest when taken in connection with the electrical apparatus shown in cuts 1 and 2.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

ELECTRIC PIG IRON IN WAR TIMES

By Robert Turnbull.*

[Abstract.]

The influence of war-time conditions on electric pig-iron production is studied, and the reasons discussed why electric pig iron is not being made at present in North America, although the profits would appear attractive.

The manufacture of low-phosphorus pig iron (now selling at a very high price) by melting steel scrap in an electric furnace is described, and its commercial possibilities discussed, with particular reference to post-bellum conditions. The addition of steel scrap to blast furnaces and electric pig-iron furnaces to increase their output is also described, as a procedure which is profitable under present conditions.

The abnormal high prices which are at present being paid for iron and steel products, and which have been ruling for the last eighteen months, have seemingly failed to stimulate or encourage the manufacture of pig iron from ore in the electric furnace. Although from $50.00 to $80.00 per ton is now being paid for the different grades of pig iron, no progress has been made in the United States or Canada in the production of electric pig iron from ore, and it may safely be said that outside of Sweden and Norway the production of pig iron from ore in the electric furnace is practically *nil*. That pig iron could be made from ore in the electric furnace under present conditions, and at the same time yield a handsome profit, there is not the slightest doubt, but there are many difficulties in the way of the prospective manufacturer and one of the most serious is the shortage of power.

* Electric Furnace Engineer, Welland, Ontario, Canada.

In the early part of 1906, when the smelting of pig iron in the electric furnace was undertaken by the Canadian Government, the report issued some time afterwards claimed that the results of the experiments had proved that four tons of pig iron could be produced per horse-power year, in other words, this amount of iron could be produced with an expenditure of about 1,650 kw. hours, whereas the present Swedish practice, where large furnaces are used, shows that from 2,500 to 3,000 kw. hours is required to produce one gross ton of grey iron.

A few years ago the writer made some experiments with a single-phase furnace using about 650 kw., the ore used being a hematite containing from 50 to 52 percent metallic iron. This furnace was operated for several weeks and the expenditure of energy per gross ton of pig iron produced was 2,300 to 2,400 kw. hours. It will therefore readily be seen that to produce any considerable quantity of iron in the electric furnace, large amounts of power will be necessary, and this, coupled with the fact that such an enterprise could only be operated under existing conditions, which are liable to change at any moment, has no doubt deterred investors from risking the capital necessary for such an undertaking.

The writer's own experience has not gone to show that any advantage has been gained either by lowering the expenditure of power or decreasing the gross cost of the product by the use of the shaft furnace, such as is used in Sweden, for unless the gases are utilized the shaft furnace becomes a complicated, expensive and unnecessary piece of apparatus. A well-designed ordinary electric smelting furnace with partial roof and automatic charging from overhead hoppers, will produce a cheaper pig than the shaft furnace, and the first cost of plant would be considerably less.

With the war, however, a new industry has been created for the electric furnace, which owing to its practically low initial cost in plant and low power consumption, bids fair to become very popular, and that is the production of pig iron from shell turnings and shell scrap.

As the specifications for steel to be used in the manufacture of shells call for low phosphorus and sulphur, it is possible in the electric furnace to produce a low phosphorus pig iron from such

scrap at a figure which, given the present selling price of low phosphorus pig iron, yields a handsome profit, while very little refining is necessary. Any type of steel furnace can be used for this process, and it is not even necessary to have a furnace of the tilting type when little refining has to be done, as by special manipulation of the slag in the furnace, a certain amount of phosphorus can be removed, and sulphur is always to some extent eliminated by the basic slag employed.

The possibility of this process was first brought to the writer's attention in the fall of 1916, and experiments were immediately undertaken in a small furnace at Orillia, Ontario. These experiments having proved the process to be a feasible one, a new single-phase furnace was built and has been in successful operation since January of the present year. From 6 to 7 tons of low phosphorus pig iron is being produced per 24 hours, the amount of power used on the furnace being about 250 kw.

Furnaces for this work have since been installed at St. Catharines, and Collingwood, Ontario, also at Shawinigan Falls, Quebec, and a number of furnaces are now operating in the United States.

At St. Catharines, the furnace is of 6-ton capacity, three-phase, operated by Packard transformers of 1,200 kw., but only 700 kw. is being used at the present time, owing to the shortage of power. The daily output is around 20 tons. It is estimated that the production will attain 35 gross tons with the full power of 1,200 kw. The operation is in many respects similar to the making of steel in the electric furnace, the refining process for the elimination of phosphorus being somewhat different. The charge consists of shell turnings, the necessary carbon base, which may be charcoal, coke or coal, ferro-silicon and lime, manganese is not added, as care is taken in the process to conserve the manganese already contained in the turnings. The furnace is of the fixed type, with a carbon bottom and silica brick lining, there are two charging doors and one tap hole, similar to what is used in open-hearth furnaces. The furnace is controlled by automatic regulators, which eliminate to a great extent manual labor on the furnace, besides giving a much steadier load than is possible by hand control. The product is cast in sand, the pig beds being placed

about 30 feet from the furnace. In our Orillia plant the iron is cast into iron moulds, which make a better looking pig than sand casting. The return in iron as compared with the scrap charged is about 95 percent, and as about 5 percent is added to the iron in the form of carbon and silicon, the total loss is about 10 percent of scrap charged. The pig produced is very tough and the analysis is fairly uniform, as can be judged by the following table of consecutive heats:

Heat No.	Silicon	Sulphur	Phosphorus
237	1.45	0.025	0.025
238	1.83	.029	.031
239	2.82	.030	.033
240	2.11	.029	.027
241	1.88	.025	.027
242	2.49	.027	.027
243	1.21	.035	.040
244	2.35	.030	.030
245	1.25	.029	.031
246	1.55	.027	.027
247	1.59	.028	.031
248	1.50	.025	.029
249	2.02	.029	.032
250	1.50	.032	.038
251	1.41	.029	.033
252	1.50	.025	.023
253	2.20	.025	.027
254	1.50	.027	.030

The carbon is very rarely over 3 percent; from this point onwards any extra carbon added is very destructive to the lining and roof of the furnace. A roof will last from 150 to 200 heats; the roof conditions are being gradually improved and 300 heats may yet be realized for one roof.

The process is not a new one, as attempts were made several years ago to make pig iron from scrap steel, but the cost was found to be prohibitive. The writer also made some experiments in operating the pig-iron furnace for ore, before mentioned, by mixing 50 percent each of scrap and ore in the charge. The result was an increased production from about 6 tons on ore alone to 11 tons with the mixture. The same amount of power was used in both cases.

In passing, it might be said that some of the blast furnaces in

the States are now mixing considerable quantities of steel turnings with the ore, in order to increase their output.

Whether this process will be feasible after the war and when normal conditions are again with us is not a settled point. The cost of production is at present high, and would be prohibitive under normal conditions. It must be remembered, however, that costs today are possibly fifty percent higher than they were three years ago, and it is the writer's conviction that provided future experiments find a means of eliminating phosphorus from the iron without removing the slag, the process can be profitably operated in normal times, at least in Canada, where low phosphorus pig iron is always a dollar or two higher than in the United States.

In conclusion, although a little foreign to this paper, the writer would like to call the attention of all those interested in steel furnaces to the roof coolers, which are placed around the electrodes in order to keep the bricks at that point as cool as possible. Recent practice at St. Catharines has shown that the life of the roof can be prolonged materially by correct application of these roof coolers, especially the ring of bricks around the electrodes. These coolers should be made to the exact form of the roof, and should rest on the bricks which form the ring around the electrode, and not on any packing between these bricks and the cooler. They should not be flanged, but cast to a special form, so that the cooling water will be at an equal distance from all outside surfaces. The writer will be glad to give any further particulars to members who may be interested.

DISCUSSION.

ROBERT TURNBULL: The number of kilowatts per net ton was slightly over 700. That was measured on the peak; the integrated k.w. were a little less than 550. The load factor for the month was 58 percent. That was with two shut-downs for relining, and putting a new roof on the furnace, also for repairing and adding new rings around the electrodes.

The last fifteen days of last month produced iron carrying sulphur 0.019 and phosphorus 0.026. We are getting better results all the time in the way of refining metal.

Then I spoke of the carbon, about the difficulty of getting it over 3 percent. We have had it over 3½ percent, but still we nearly lost one roof in the process.

Regarding the roof cooling rings, many of you know of the necessity of keeping the roof brick in good condition, as the gases escape there and it is hard to keep those brick from melting. Roof coolers have been used for many years; at first they were flanged, and where the water circulated the brick were cool, but at the flange the cooling effect on the roof was not appreciable. Later we have been using coolers where the water is conducted to every portion of the cooler, so that the total surface is cooled at every point, and we have been able to get ten days' more life out of our roofing. The last roof we had gave about 260 heats.

PRESIDENT C. G. FINK[1]: Of course the secret of success of pig iron as made in the electric furnace, as compared with the old process, is cheap power. Mr. Turnbull has referred to water power in Canada. The Department of the Interior of Canada has submitted to us a brochure on the water powers of Canada, and any member wishing a copy can secure it from the Department of the Interior, by addressing Hon. W. J. Roach, Minister, Ottawa, Canada.

J. A. MATHEWS[2]: I have had no experience in the manufacture of pig iron, but one thing occurred to me in reading Mr. Turnbull's paper, in reference to the raw material he proposes to use. Why does he put those turnings back into pig iron to be re-made into steel?

R. TURNBULL: There are two replies to that question. One is that in Canada at present we have a serious shortage of low-phosphorus pig iron. We have in Canada about twenty acid open-hearths, varying in capacity from 15 to 35 tons. It has been very hard to get low-phosphorus pig iron from the States; and it will be harder in future. We are obliged to show that any iron secured is going to be used for war purposes, and unless this can be shown, the entire importation will be shut out. If shut out, we can only get low-phosphorus pig iron by this process. The blast furnace men in the U. S. A. have been using 5,000 tons of

[1] Director of Research, Chile Exploration Co., New York City.
[2] Manager, Holcomb Steel Co., Syracuse, N. Y.

our shell turnings per month, and we are obliged to give them that to get the other in return. Under present conditions we get a good return, using our process, for the money invested.

LAWRENCE ADDICKS[3]: Regarding the failure of roof rings on copper reverberatory furnaces, we have been putting alternate thick and thin rings; the thin rings allow radiation and the thick rings hold the furnaces together. I believe they have gotten three or four times the life out of them compared with where they were uniform. Those are wide furnaces, thirteen or fourteen feet wide.

E. F. CONE[4]: I want to ask Mr. Turnbull if any of the iron has been tested physically, in the shape of castings or as test bars. I have seen tests of such iron from bars one inch square, which, from the average of a series of tests made at Columbia University, showed a tensile strength of 45,000 lb. per square inch with the modulus of rupture at 25,000 lb. This is double the value of ordinary gray iron. I understand the structure is quite different from that of low-phosphorus blast furnace iron.

I am interested in his statement that the kilowatt-hour consumption in his practice is 700. The claim has been made to me, that such iron could be made with a consumption of 300 to 500 kw. hr. per ton. I very much doubt the 300 figure. I was interested to note that it was 700 in Mr. Turnbull's case.

R. TURNBULL: We have seen only those tests which the steel companies have been getting from our metal. It is very hard to break under the hammer; it is much tougher than blast-furnace iron.

Regarding power consumption, I have not taken it on separate heats. I do not think we could get down to 300. I believe we could get 500. That is not measuring on the peak. If it were done at 500, on integrated power, it would be good practice.

J. A. MATHEWS: I do not quite yet follow the reason for Canada converting scrap into good low-phosphorus pig iron, or why they would not be better off if they converted all of their turnings directly into electric steel for munitions, rather than pass it back through pig iron to be returned to the open-hearth furnaces.

[3] Consulting Electrometallurgist, New York City.
[4] Associate Editor of *Iron Age*.

R. TURNBULL: Dr. Mathews knows what it would be to turn the acid open-hearth furnaces to basic. I think the conditions of basic practice are bad enough without putting on more basic furnaces when material for basic lining is so scarce. We have to turn out every ton of steel we can produce. I do not think I could advocate his scheme.

F. A. J. FITZGERALD[5]: Mr. Turnbull spoke of the importance of electrode cost. Can any figures be given of the electrode consumption per ton of pig iron?

R. TURNBULL: About 45 to 46 pounds.

H. E. RANDALL[6]: Answering the question as to why electric furnace plants are started to manufacture cast iron, rather than steel, I would say that the answer is that it requires a much smaller capital expenditure for a given net return, and thus one gets his money back much faster by manufacturing cast iron, rather than manufacturing steel from scrap.

With respect to Mr. Turnbull's statement about power, I cannot let it go unchallenged. He confuses the whole of Canada with the Province of Ontario. In Quebec Province we are well able to supply power, and, on a long term basis, the price would be somewhere around 60 to 75 percent of his figures.

E. F. CONE[7]: I intended to ask Mr. Turnbull what the percentage of combined carbon in the pig iron is, as he makes it?

R. TURNBULL: I think the combined carbon is about 1.6. Owing to the low carbon the grain is very small as compared with ordinary 3½ percent carbon iron.

MR. TURNBULL (*Communicated*): I find I misunderstood Dr. Mathews' second question, as my answer does not correspond to the question as put. I mentioned in answer to Dr. Mathews' first question that we had in Canada about 20 open-hearths operating on the acid principle, which it would be safe to say would produce at least 1,500 tons per day of steel. The quantity is in reality much greater, as there are more acid open-hearths than the figure given above. About 10 percent of Bessemer iron and 10 percent of low-phosphorus iron is used in the charge for these

[5] FitzGerald Laboratories, Niagara Falls, N. Y.
[6] The Shawinigan Water and Power Co., Montreal, Canada.
[7] Associate Editor of *Iron Age*.

furnaces, which, taking 1,500 tons as the production, means 150 tons of low-phosphorus iron per day. To produce this latter would require at the most 5,000 kilowatts. But were the total 1,500 tons converted direct into steel in electric furnaces, 50,000 kilowatts would be required for this purpose, which aside from the question of the shortage of power, would mean an investment of probably three millions of dollars in plant. On the other hand, it would be necessary to close down all the acid open-hearth furnaces, which would ruin that industry. Today (February 22, 1918) there is not one pound of low-phosphorus pig iron being imported from the States, and these acid open-hearth furnaces are absolutely dependent on the pig iron made from shell turnings, to continue operating.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

THE ELECTRIC FURNACE IN THE DEVELOPMENT OF THE NORWEGIAN IRON INDUSTRY

By Haakon Styri.*

[Abstract.]

The development of the Norwegian iron industry is briefly reviewed, followed by a detailed account of the introduction of electric steel furnaces and electric pig iron furnaces. The effect of present abnormal war conditions on the expansion and future of the industry is discussed and the commercial relations regarding supply of ore, fuel and steel scrap are elaborated. The development of casting, rolling, and other manufacturing plants is outlined; also the conditions controlling the present investment of capital in these industries. Regret is expressed that the Government is favoring non-electric plants, while private capital is more inclined to the development of electric methods.

At the end of the sixteenth and the beginning of the seventeenth centuries, Norway had a comparatively well-established iron industry, which, after a declining period in the hard years of war following 1800, again revived and flourished until the middle of the last century. Eighteen iron works were in operation, and many prominent families made fortunes at that time. It is probable that from the same period originated the name "Norway Iron" for high-quality wrought iron.

With the common use of coal and coke in the iron works, and with constantly increasing demands for wood for lumber, pulp, and cellulose, the competition for our charcoal became severe, and a constant decline in the iron industry followed, until, at the

* Metallurgist, Hussey-Binns Steel Co., Charleroi, Pa.

end of the century, only one charcoal furnace, at Naes, was in operation, utilizing its product for crucible steel.

A turning in the tide again seemed to come with the steel-casting industry. An acid open-hearth, heated with generator gas, was built at Kristiania Staalverk, and a Tropenas converter with cupola outfit at Strömmen Staalverk. A third plant made a good profit by welding together plate scrap to billets, which were rolled and forged.

The application of electric energy in iron and steel making gave a new impulse to the iron works, supported probably by the competition between owners of water falls who wanted to find some use for their power.

Jössingfjord Manufacturing Company, in 1909, built an induction furnace of the Hiorth type,[1] and produced some excellent steel from high-grade raw material, but without commercial success. This was the only Hiorth furnace in operation until recently, when, it is said, another furnace was built by Kværner Bruk.

More interest centered in the electric production of pig iron, after the promising reports from the operation of the "Electrometall" shaft furnace at Domnarfvet and Trollhättan. The Electrometallurgical Committee, appointed by the Norwegian Government in 1907, reported favorably on the prospects, and with nation-wide support, the concern, Hardanger Jernverk, was formed at Tysaa.

The type of furnace adopted here was the "Electrometall," but with modifications in design, which, after the few experiments in Trollhättan, were considered necessary when coke was used for reduction. One furnace was built and put in operation in 1911, and another built before results from the first were obtained. The operation was not successful. The charge had a strong tendency to hang, caused partly by the narrow neck, and, partly as later investigations have indicated, by the limestone, which fell into powder when burned, and by the precipitation of carbon in the briquettes, causing many of these which were of a loose structure to burst.

After some time it was also found that the electric arrangement was bad. The voltage range, from 50 to 78, was not sufficient.

[1] Trans. Am. Electrochem. Soc., **20**, 293 (1911).

At times, a higher voltage was necessary to increase the energy consumed, particularly after setting of a cold charge in the furnace. The leads to the furnace and connections to the electrodes were so arranged that a power factor of only 60 percent was obtained. This was, however, improved upon in the second furnace, which gave a better power factor, but, on account of the cost, the transformers and voltage regulation could not be changed. On the average, therefore, only part of the rated load could be held. As a result, the charge went cold, the tapping was often difficult, and the product was not uniform. To improve the running, gas circulation was abandoned, with an increase in the electric energy- and coke-consume.

In the charge, it was intended partly to use ore concentrates, which were roasted and agglomerated in a Peterson roasting furnace before charging. The roasting was good but the agglomeration very poor. More expensive lump ore had to be provided. Freights increased; coke increased in price, and was probably not of right quality. After a year of unsuccessful operation, the plant was closed down. The capital invested was not large enough to overcome the pioneering difficulties. Of course, this does not prove that coke cannot be used in the Electrometall furnace, and it is said that in later tests in one of the larger new furnaces in Sweden, where coke alone was used for reduction, satisfactory results were obtained. Some recently published reports, however, indicate that reduction with coke was tried in the Helfenstein furnace in Domnarfvet, where the energy consumed was 2,600 to 2,700 kw.-hrs., and the coke consumed 310 to 330 kg. per ton of pig. From the same source, we are informed that a Helfenstein furnace is in course of construction somewhere in Norway. Time will tell whether or not sufficient tests have been made to justify this new construction.

Before the Hardanger Jernverk started operation, experiments were made from January 1, 1910, at Notodden, with the 500 kw. furnace of the Bie-Lorentzen and Tinfos Paper-Fabric. After quite successful trials with coke as reduction material, the Tinfos Jernverk was formed, which built three 1,200-kw. furnaces of the single-phase type, with bottom electrode, rectangular chamber, and two charging stacks.

Work started in 1912. In the beginning many difficulties had to be overcome, partly on account of poor electrodes and bad castings for water-cooled collars. Many furnace details had to be changed, and the correct way of mixing and charging the material at hand had to be found, also most careful attention had to be paid to the physical properties of the coke. The product at first varied considerably in quality, but now the furnaces operate very regularly. White and grey pig iron of high quality can be produced at will, competing with charcoal iron. It may be of interest to note that most of the manganese in the ore is reduced.

The design of the furnace makes a utilization of the furnace gases mechanically difficult, and no attempt has been made to do so, neither is gas circulation used. From data published, the energy consumed per ton is 2,700 kw. hrs. for white pig and 3,000 kw. hrs. for grey pig, with 44 percent iron in the ore.

The Tinfos Jernverk has had a very successful period since the war started, and particularly since the demand for high-grade pig increased. It was practically the only domestic plant which could furnish a small part of the pig iron that was formerly imported from Sweden, Germany and England, and has thus materially helped in keeping the mechanical industries of Norway going.

A Tinfos furnace has also been built at Ulefos, using phosphoric ore from the Faehn deposits. Foundry iron is produced, which is remelted in a Rennerfelt furnace and then run into stove castings.

None of these plants have, until now, tried to convert the electric pig iron into steel; but electric steel making in the country has developed along the same lines as in other countries where electric furnaces have been adopted.

The most important concern, melting and refining scrap for tool steel and steel castings, is the Stavanger-Staalverk, which started operation in 1913. The scrap is melted in an open-hearth furnace, and refined in a 5-ton Roechling-Rodenhauser single-phase induction furnace. This plant has gradually improved its product, and now has a very good reputation for hammered bars and steel castings. The plant has been very prosperous during the last two years, and recently increased its invested capi-

tal to $1,000,000, in order to allow installation of more electric furnaces, a bar mill and electric annealing furnaces. It is also said to have discontinued the operation of its open-hearth for melting down the scrap, on account of the present prohibitive prices of coal.

A number of other small electric-furnace installations have been formed on account of the wartime profits in steel castings and special steels. A small Roechling-Rodenhauser furnace at Eureka, Kristiania, makes acid-resisting steel; at Hamar, Hougsund, and Drammen, cast steel is made in Rennerfelt furnaces, and at Kongsberg and Naes tool steel is made in the same type of furnace.

It will be noted that most of the electric furnaces built in Norway are of the Rennerfelt type. The reason for this is that it is difficult to get material from countries other than Sweden, and the furnace is very well adapted to the remelting of scrap for cast steel in small quantities.

There is very little in the feature claimed as specific to this furnace, *viz.*, that the arrangement of electrodes serves to throw the arc against the bath. Anyone will find that an arc will play against the molten charge when, with single- or poly-phase current, the electrodes are placed at an angle to the charge. The arc will follow the path of least resistance, that is, where the gases are hottest between the electrode-ends and the bath. When starting to melt down cold scrap, the arc is not more steady than it is, for instance, in a 3-phase Heroult furnace, because of the cooling of the air from the charge with the consequently unsteady air currents. The lining is very much exposed to radiant heat, and the uneven heating of it has been the result of faulty shape of the furnace body, which has been improved in recent designs, approximating the well-known Stassano furnace.

The Heroult furnace is not, at present, in use in Norway for steel making, but it will probably be seriously considered by a new larger concern. The excellent shape of the furnace body, which facilitates repair, and the simple arrangement of electrodes, are of great advantage. Many details in the furnaces built by the U. S. Steel Corporation could certainly be improved upon. There ought to be sufficient room under the electrodes for me-

chanical charging, an essential point in the economy of operation. In a larger plant the electrodes should be arranged on the side, to allow a continuous charging platform for a battery of furnaces. Attention should also be given to the possibility of the freezing of the water in cold weather. All pipes, therefore, should be easy to drain. The device for lifting the doors, and the manner of tightening them should be improved.

Much of the successful operation of the furnace depends on the kind of scrap that is used. To avoid losing time in melting, the complete charge should go into the furnace from the start, and it is very important that the smelter pay attention to his lining when charging, as he can protect or cut the lining according to the way different material is placed.

In larger furnaces, from 16 to 20 tons, graphite electrodes are probably better than carbon electrodes, because it is difficult to handle the large-sized carbon electrodes, particularly when they break off in the furnace during operation. Besides, if private information is correct, they are more economical in use, at least when applied on molten charge.

The refining facilities in the Heroult furnace are very good. There is no difficulty in getting an oxidizing slag. The reducing action of the electrodes will not overbalance the oxidizing influence of the incoming air, which effect can be increased by roll scale and ore. When making high-carbon steel, in particular, it is of advantage to dephosphorize at the lowest possible temperature, in order to enable the greater part of the carbon to remain. In the preliminary testing, the manganese content will indicate the amount of phosphorus, when the content of both in the material charged is known. For desulphurization the furnace is ideal, because the slag can so easily be kept reducing by throwing in coke or coal, and, if necessary, ferro-silicon, whereas the temperature of the slag can be kept high.

A common disadvantage in the arc furnaces is that so much smoke is developed, which, in some places, has even resulted in strikes by the workmen. This fault is not shared by the induction furnace.

As to the facilities for refining steel in the induction furnace, the elimination of phosphorus is easy, and the elimination of sul-

phur can also be done well, but the metallurgical conditions for the latter are not as good as they are in the arc furnace.

The low power factor in induction furnaces, which necessitates a low frequency when the synchronous condenser is not used, has, in other countries than Germany, hampered their common use, at least where power must be taken from an outside source. Where the power plant is constructed entirely for a steel plant, much of the self-induction can be taken care of by over-exciting the field of the generator, as has been stated in the Transactions of this Society by Strong and FitzGerald. Roechling-Rodenhauser tried to increase the power factor by introducing secondary pole plates in the furnace, but, in some places, at least, these were abandoned because they were more of a nuisance than a benefit. The Roechling-Rodenhauser 3-phase furnace, with three heating channels, has not been successful. It seems better to apply the 3-phase current in Scott connection in a 2-channel furnace, which gives the simplest melting hearth.

Where furnace installations are contemplated, the choice of furnace type will, to a great extent, depend more upon the cost of installation and royalty than upon the kind of work that is to be done and the ease of operation. It is well to remember, in this connection, that the first and principal Heroult patents have now expired.

In late years, in Norway, there has been a great desire to get iron and steel works which could produce shapes, plates, and other commercial steel in order that the country might be more independent of imports. During the present war this need has been bitterly felt, as it is extremely difficult to get commercial steel from the belligerent countries, which formerly furnished it all. Many private corporations have investigated the possibilities of starting a modern steel plant, and much capital was expected to back the plans if they could be satisfactorily worked out, but the present soaring prices of all new construction has put a stop to further important development. Another reason for little progress in the plans was the difficulty in working the different local interests together into a national interest, and the

intervening of the Government has not improved the conditions. It is of importance, however, that Government interest in the development has been created. The immediate result of this is that, as a strictly war measure, a premium on the production has been granted to Kristiania Spikerverk for building a second open-hearth furnace and a small bar mill, and the extra funds necessary to build a new cupola and Tropenas converter has been voted to Strömmen Staalverk. It is rather discouraging for people interested in the electric iron industry in Norway to find that the Government considers it more opportune, economically, to support open-hearth and Tropenas converting and not electric steel melting, when private interests find it best to work along the latter line; but the reason is probably that, in the present emergency, it is easier to get building material for open-hearth furnaces and converters, and wood and charcoal can be substituted for coal and coke.

A survey of the possibilities of the future development of the iron industry in Norway may be of interest.

The country is long and narrow, mountainous, and cut by many fjords. It takes a 12-knot steamer about six days to go around the coast from Kristiania to the Russian border.

The only coal deposit is on Andöen, one day's voyage from the Russian border, and it is of no practical interest. The coal deposits on Spitzbergen, which is a two days' voyage north of Norway, are mostly owned by Norwegians, and are calculated to produce 400,000 tons annually from the year 1920, which production probably can be increased to 800,000. The Spitzbergen coal is excellent steam coal, but not well suited for coking. As the yearly import in Norway of coke and coal is 3,000,000 tons, it will be seen that the supply and price depend entirely on England, Belgium and Germany.

The main ore deposits are all in the northern part of the country, and the most important ones are low grade, with 30 to 35 percent iron. Only a few small deposits can furnish 50 to 55 percent iron. Therefore, the ore is usually concentrated. The principal concern, Sydvaranger, situated near the Russian border, has planned an export, after the war, of 900,000 tons of concentrate a year.

Next to Sydvaranger, we have in the Ofoten district a number of deposits, a few of which give 50 to 55 percent ore. Further south, two and one-half days' voyage from the Russian border, we have a big deposit of low-grade ore in Dunderland, where nearly $10,000,000 was invested in a company which intended to use the Edison method of concentration, but which proved unsuccessful. The reconstruction of the concern, using other methods for concentration, has been tried a number of times, but so far without definite results.

Other deposits are around Trondhjemsfjord and Romsdalsfjord, all low-grade ore. From here and south to Kristianssand, the deposits are of little commercial value. The deposits in the eastern part of the country, which, however small, furnished the lump ore for the many small charcoal furnaces in operation in the middle of the last century, are at present still important, as they supply the ore for Tinfos and Ulefos Jernverk.

North of Trondhjem there are some good-sized waterfalls; a few of them are not very far from ore deposits. Water power is most abundant, however, on the western coast, where there is little ore. In the southeast, water power is more expensive, and ore is scarce, but in this district, around the Kristianiafjord, the population is more dense, and two-thirds of the total import of iron and steel is consumed there. The rest of the imports are, for the greater part, taken by the west coast, and only a little goes north of Trondhjem.

It will be seen that the conditions do not look promising for the production of steel on a larger scale in Norway.

The operation of a coke blast furnace would demand the import of lump ore from Sweden, Spain or North Africa, besides the fuel from England, and the same would, to a less extent, hold true for a large scale production of electric pig iron.

For the conversion of pig iron to steel there would be little difference in the cost whether this was done in the open-hearth or electric furnace.

The freight on some or all material used would increase the final cost considerably.

The conditions are no better, when we remember that Norway

and England are the only iron-producing countries which have no duty on imported iron.

Better economical success could apparently, at least before the war, be expected for a concern remelting scrap in electric furnaces, as the scrap was considerably lower in price than pig. The basic open-hearth would certainly melt the scrap somewhat cheaper than the electric furnace, but when the capacity of the latter is increased it is probable that the steel from an electric furnace using cold scrap would not cost more than $1.00 to $2.00 per ton more than the open-hearth steel, when the same quantity is produced, and the better quality of the electric steel would balance the difference. Increasing coal prices would improve the prospects for the electric-furnace melting. The difficulty would be to get the scrap. The total annual import of steel into Norway is about 200,000 tons, from which 12 to 15 percent is returned as scrap. The scrap from all parts of the country would therefore not amount to more than 30,000 tons, and most of this is already disposed of by the plants in operation. A large concern must, therefore, depend entirely on the supply of scrap from neighboring countries, with additional freight expense and at prices in competition with the foreign buyers.

The prospects can, of course, change. It is possible that concentrate can be used direct in an electric reduction furnace, at least where little attempt is made to utilize carbon monoxide in the reduction of the ore, by using the gas instead for heating pig-iron mixers, where the main decarbonizing could be done, for melting of scrap, and for heating of by-product coke ovens. The gas from the coke ovens could be sold to the city. The steel product could be finished in an induction or arc furnace.

Another method is now on trial, basing the operation on the direct use of low-grade ore by the Edwin-Westberg-Gröndal process. Interested Norwegians and Swedes have secured $150,000 for the purpose of testing the method on a commercial scale. The low-grade ore is crushed according to the structure, and is treated in the furnace with reducing gases which are heated outside in a high-tension arc furnace, similar to the one used for the manufacture of saltpeter. The reduced iron is afterwards separated magnetically from the rock, and the iron powder melted in an electric furnace.

We have seen that the successful iron and steel works in Norway have based their production on high quality of product. The manufacture of structural steel is not, and probably will not be tried in the near future, on account of the competition from foreign countries.*

What Norwegians want and look forward to is the time when the country can again furnish iron and steel which comes up to the old standard of Norway iron.

DISCUSSION.

J. W. RICHARDS[1]: Has there been any use in Norway of the iron sponge that was being made in Sweden at Hoganäs?

HAAKON STYRI: There have been made some tests of iron sponge in making electric steel, with very good results. A very high quality of steel was made.

F. A. J. FITZGERALD[2]: I think I can answer Dr. Richards' question in part: A few years ago we got at the laboratory two or three tons of sponge iron made in Sweden and got some excellent results melting it down to make steel in a small electric furnace of the Heroult type.

When I visited Sweden some years ago and heard about the use of iron sponge there, I was told that the losses by oxidation when melting down in open-hearth furnaces were large.

Referring to Mr. Styri's remarks as to the use of graphite electrodes in 20-ton electric steel furnaces: Have there been any extensive experiments made with the use of amorphous carbon electrodes? In general I prefer the use of amorphous carbon electrods, in the Heroult steel furnace, to graphite, because the loss of heat by radiation to and from the roof is less with the former.

HAAKON STYRI: I referred to some American practice. In some of these American steel mills I have visited, graphite elec-

*Kristiania Staalverk is at present (January) increasing its capital to $5,000,000 for the building of a plate mill (production 50,000 tons) and necessary open-hearth and electric steel furnaces, and possibly later electric pig-iron furnaces.

[1] Professor of Metallurgy, Lehigh University.

[2] FitzGerald Laboratories, Inc., Niagara Falls, N. Y.

trodes were used in place of amorphous carbon electrodes. I have no personal experience in that matter.

(*Communicated Nov. 20, 1917*): According to recent information, a loan, free of interest, has been granted by the Storthing to Hamar Mekaniske Versted, to build an electric steel plant.

Tinfos Jernwerk, making electric-furnace pig iron, has since the first of this year had serious difficulties in getting sufficient raw material for their operation. The raw material has also been of very poor quality, which has had a bad effect on the product.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

A RESISTANCE FURNACE

By Frank Thornton, Jr.

[Abstract.]

A discussion of the heating of furnaces by resistors placed inside the furnace, either on the hearth of the furnace or on a shelf around the walls. The scope of operations suitable to such furnaces is mentioned, and the nature of the resistor material, its purity and its electrical characteristics are discussed at length. Experiments were made to determine the suitability of various materials, and best results were obtained with silicon carbide blocks, bricks or rods. Satisfactory running with temperatures up to 1,200° C. was obtained.

The design of an electric furnace for heating steel to forging temperatures and for other metal-heating operations has been given a great deal of study during the past few years. Increases in the cost of all kinds of fuel and decreases in the cost of electricity available in the industrial heating applications have directed attention to the possibility of electric furnaces. In addition to the matter of fuel costs, there is also to be considered the possibility of increased speed, reduction of material losses, greater uniformity and higher average value of product. It is not the intention here to discuss the economics of the industrial electric heating furnace, but rather to communicate the experience and results obtained in a series of investigations on this subject.

It is assumed that a furnace construction suitable for heating steel commercially to forging temperatures, if successful, will also be suitable for a wide variety of other heating applications involving the same and lower temperatures. The temperature at which the steel must be removed from the heating furnace depends on

the type of forging operation, ranging from about 1,000° C. for ordinary dressing and shaping work up to 1,200° C. or over for high-speed drop-forge work.

Other applications of such a furnace would be:

Heat treating steel parts.
Hardening dies and tools.
Tempering dies and tools.
Annealing steel, brass, copper and other metals.
Heating copper, brass and other metals for rolling.
Melting copper, brass, etc.
Firing ceramics.
Firing enamelled ware.

To be commercially successful, such a furnace should require only infrequent repairs and when these are necessary the labor and material required should be inexpensive. This means that the resistor, refractory materials, and terminals should show a low rate of deterioration under service. Needless to say, domestic materials should be used throughout if possible. The regulation of the furnace should be automatic, or so nearly automatic that only very simple manual operations are necessary in order to maintain the desired regulation.

For these purposes it is necessary to eliminate from consideration at once all types of construction using metallic resistors, on account of the high temperature at which the resistor would be required to operate. There are no low-priced base metals which can be operated continuously with any degree of success for furnace uses above 1,000° C. This practically limits consideration to some form of non-metallic resistor. The material which is naturally suggested first for consideration is some form of carbon or graphite. Furnaces using this class of materials have been experimented upon for several years and some are now in use for lower temperature applications. Some information has been published by Dr. Alcan Hirsch (Carnegie Scholarship Memoirs of the British Iron and Steel Institute, 1914) and others, but no detailed operating data on actual installations have been available. Several attempts have been made under the writer's directions to apply

this construction for forging work, and a description of some of this work may be of interest. It was decided to erect a furnace or a series of furnaces and determine by actual test the design,

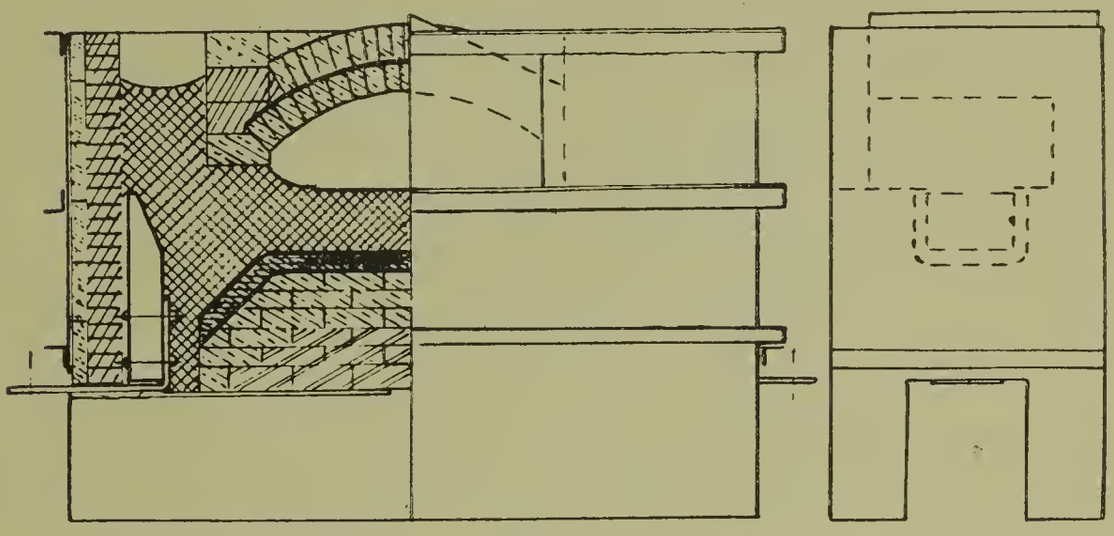


FIG. 1.

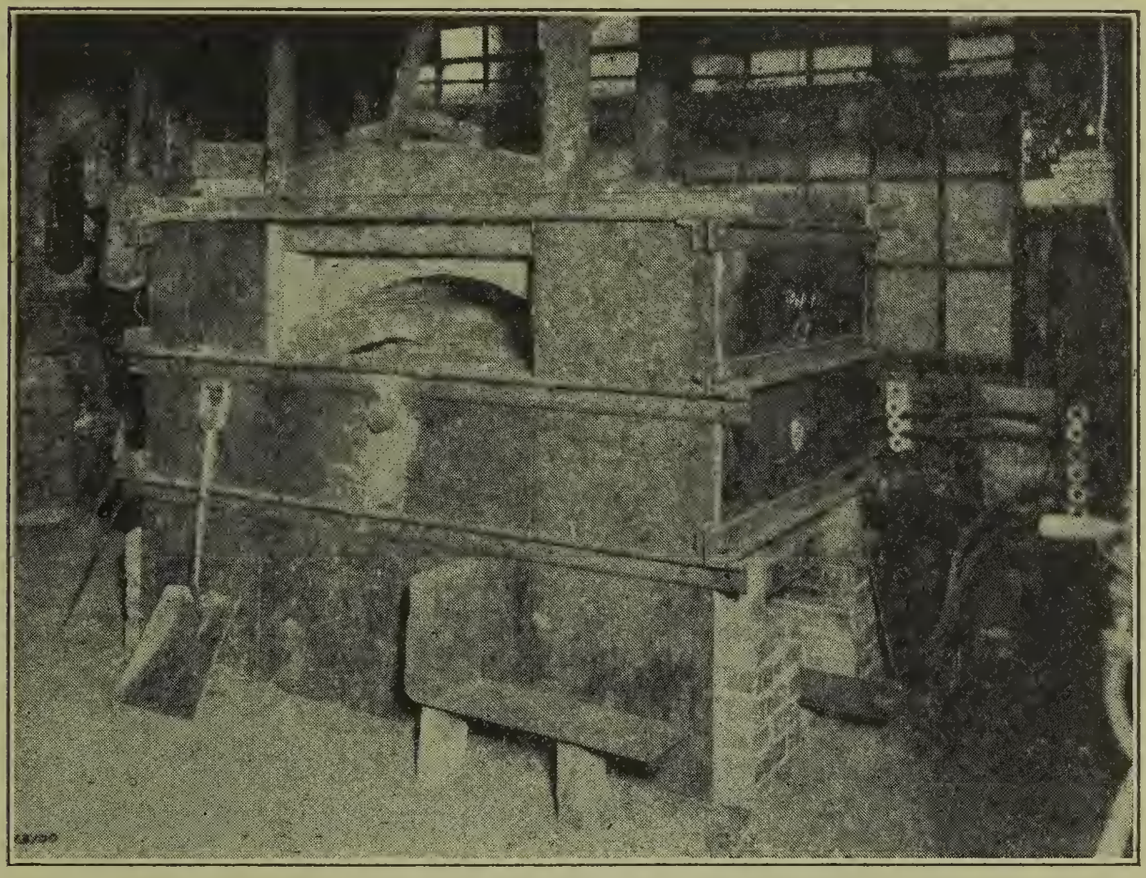


FIG. 2.

requirements, and also the limitations of application of the granular carbon, graphite, or coke, bed construction.

Working along this line, an experimental furnace was constructed as shown in Figs. 1 and 2. The chamber was approxi-

mately 36 in. (92 cm.) wide by 24 in. (61 cm.) deep by 10 in. (25 cm.) maximum height in the center of the arch. The resistor bed was approximately 9 in. (23 cm.) wide by 8 in. (20 cm.) deep, and extended across the hearth about the middle of the furnace. The terminals were located at either side of the hearth at the ends of this resistor bed, and consisted of hoppers in the brick work of the furnace which could be filled with granulated carbon, graphite, or other material, as desired. In the bottom of each hopper was a solid block of graphite to which was attached a heavy iron plate, which extended through the brick wall and served as a terminal lug for making the electrical connection. The cross section of this hopper was several times that of the resistance bed within the furnace, and it was filled with granular material of as low electrical resistance as was obtainable. The idea of this construction was to interpose between the resistance bed and the terminal blocks a mass of electrically conducting material which would also be a thermal insulator and thus avoid overheating of the main terminal blocks. Instead of the expensive terminal block being gradually burned away, there would thus be a consumption of the less expensive granular material, which could be readily replaced through the openings in the tops of the hoppers. This furnace was built and tested, and the performance of the terminal construction was as anticipated. The granular material in the hoppers was consumed very slowly, probably at a rate of not more than one or two pounds per day. The hopper could be filled to the top, and needed no attention for several weeks other than an occasional tamping. The resistor material could be replenished either through the door of the furnace or from the terminal hoppers. It required 90 volts for starting and 30 to 40 volts to maintain the furnace at 950° C. When pushed in continuous heating of steel, it could be worked up to around 40 kw. No attempt was made to secure measurements of efficiency, as more concern was felt at that time for endurance. Attention was turned to the performance of the various resistor materials and refractories.

Numerous combinations of resistor materials were tried out in the furnace, the principal arrangements experimented with being the following:

(a) Granulated graphite.
(b) Granulated coke.
(c) Graphite over charcoal (see Fig. 3).

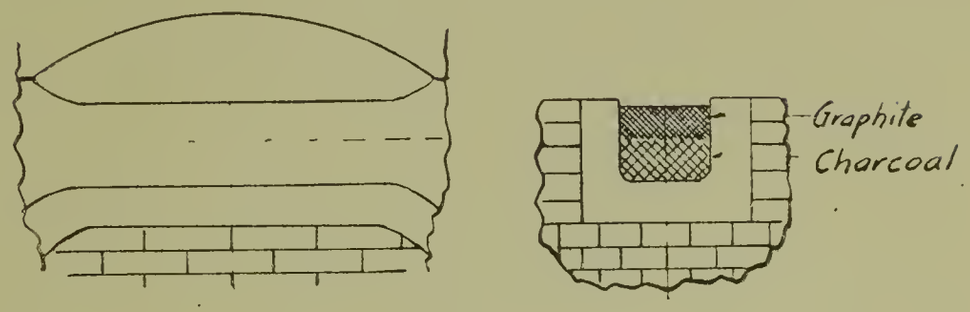


FIG. 3.

(d) Same as (c) but with carborundum blocks in the surface to aid the conduction of heat from the bottom of the resistor bed. (See Fig. 4.)

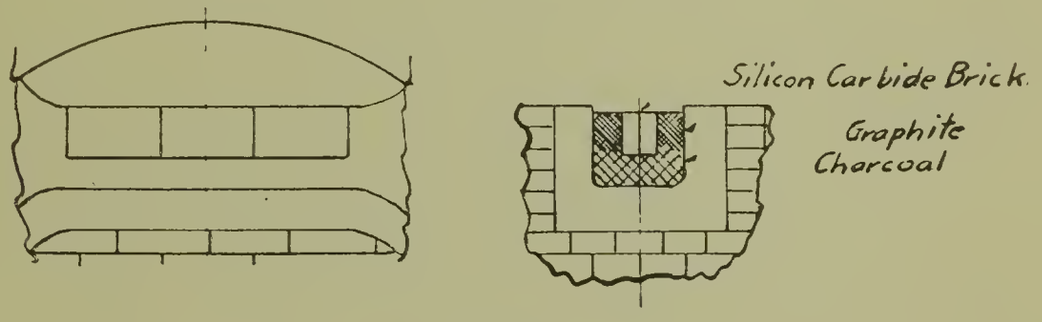


FIG. 4.

(e) Same as (c) but with solid graphite wedges in the surface to reduce the electrical resistance of the upper portion of the bed and aid the conduction of heat. (Fig. 5.)

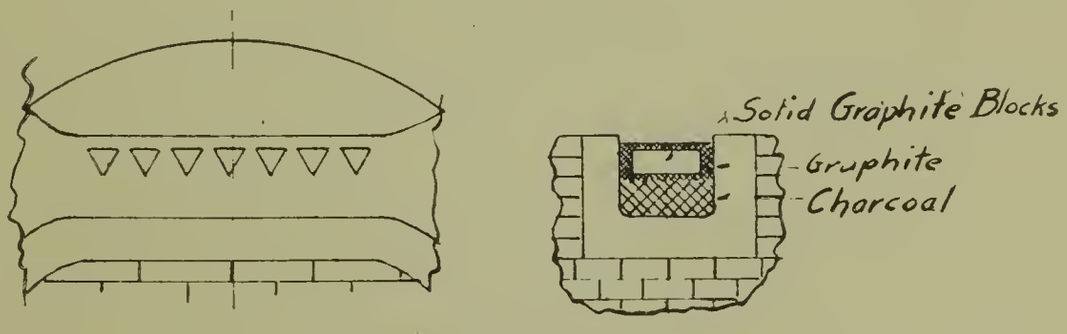


FIG. 5.

Several important conclusions were drawn from the results of the tests performed on this furnace. It was found that the resistor material must be of purest carbon or graphite. If there is any

slag or ashes left from the combustion of the resistor, it remains in the bed, so that the relative proportion of such material is gradually increased with time, causing an increase in the resistance of the furnace. The rapidity with which this action takes place is dependent upon the amount of foreign matter in the material which is added to the resistor bed, and the temperature of the bed.

If the slag or ash is fusible at the temperature of the resistor bed, a clinker will be formed which very rapidly extends throughout the granular mass, resulting in a rapid increase in resistance and consequent failure to heat. It was found that some such action as this occurred with practically all materials except the very purest graphite, and that it was only a matter of a few days or at best only a few weeks before it was necessary to close down the furnace and clean it out.

It was also found that when the furnace was maintained at a sufficiently high temperature to turn out steel heated to 1,000° C., that the temperature in the bottom of the resistor bed exceeded the safe limit of the refractory material. In order to obtain some idea of the temperature in the bottom of the resistor bed, a closed graphite tube was inserted horizontally through the front wall. The closed end of this tube was directly under the middle of the hearth at the bottom of the resistor bed. When a temperature of around 1,000° C. was maintained on the surface of the hearth it was found that the temperature at the bottom was something over 2,000° C. A study of the conditions of heat generation and transmission in this design explains how this condition is possible. Carbon has a negative temperature coefficient and the current through a mass of any form of carbon or graphite would naturally tend to concentrate in the zone of highest temperature, which is in the bottom of the bed in this case. The upper surface of the resistor bed will naturally be cooler than the lower portions of the bed because the furnace chamber is drawing the heat away from it while the bottom of the bed is thoroughly insulated. Although it would be possible to provide a path for losses in a downward direction, so as to avoid a high temperature on the lower portions of the bed, such an arrangement would prove very inefficient, excepting in special cases.

As a result of these tests, it was concluded that 900° C. was about the limit of operating temperature of a furnace using granulated carbon or graphite as the resistance material, and that there was little hope of successfully using ordinary coke or charcoal.

Another feature which these tests brought out was the importance of radiating heat equally from as large a portion of the interior surface of the furnace as possible. In heating steel in the ordinary fuel-fired furnace, heat is transferred to the work by radiation from the walls and by conduction from the hot gases. The walls are maintained at a temperature higher than that desired on the work because they also are continually swept by the hot gases. In the electric furnace we do not have this action to heat all surfaces uniformly. If heat is being generated only on the floor of the hearth, side walls and roof must obtain their heat by direct radiation. If cold pieces of metal are laid closely over the surface of the resistor hearth they will, of course, interfere with the radiation of heat to the walls and roof directly in proportion to the amount of hearth surface which they cover. In a continuously operated furnace this condition would prevent the roof of the furnace from ever reaching a temperature equal to that of the metal. Such a furnace could only be operated at a very low rate of production, which would mean a comparatively low efficiency. In order to avoid this condition, it is necessary to generate heat in the roof and side walls as well as on the floor, maintaining a high temperature on all radiating surfaces, so that heat may be fed into the work through all its exposed surfaces.

In order to prevent excessive temperatures on the refractory material it is also necessary to design the resistor in such manner as to reduce the thermal resistance of the path between the interior and the exposed surfaces. This may be accomplished in two ways, either by reducing the thickness of the bed or by using material which will have a higher thermal conductivity at the working temperature. It is not practical to operate a thin bed of granular graphite on account of the rate of combustion which takes place, and of the danger of non-uniformity of the resistance. Solid blocks of graphite or carbon have better thermal conductivity, but are objectionable because of the combustion which would undoubtedly take place on the exposed surface, and difficulty of

repair or replacement of burned portion before the loss became serious.

In view of all of the difficulties encountered with granular resistor beds, it was decided to investigate the possibility of using silicon carbide blocks or slabs for the resistance material. This material was known to resist oxidation up to an extremely high temperature, to possess considerable rigidity and good heat conductivity, and to be available in various moulded shapes. Difficulty was anticipated on account of the large negative temperature co-

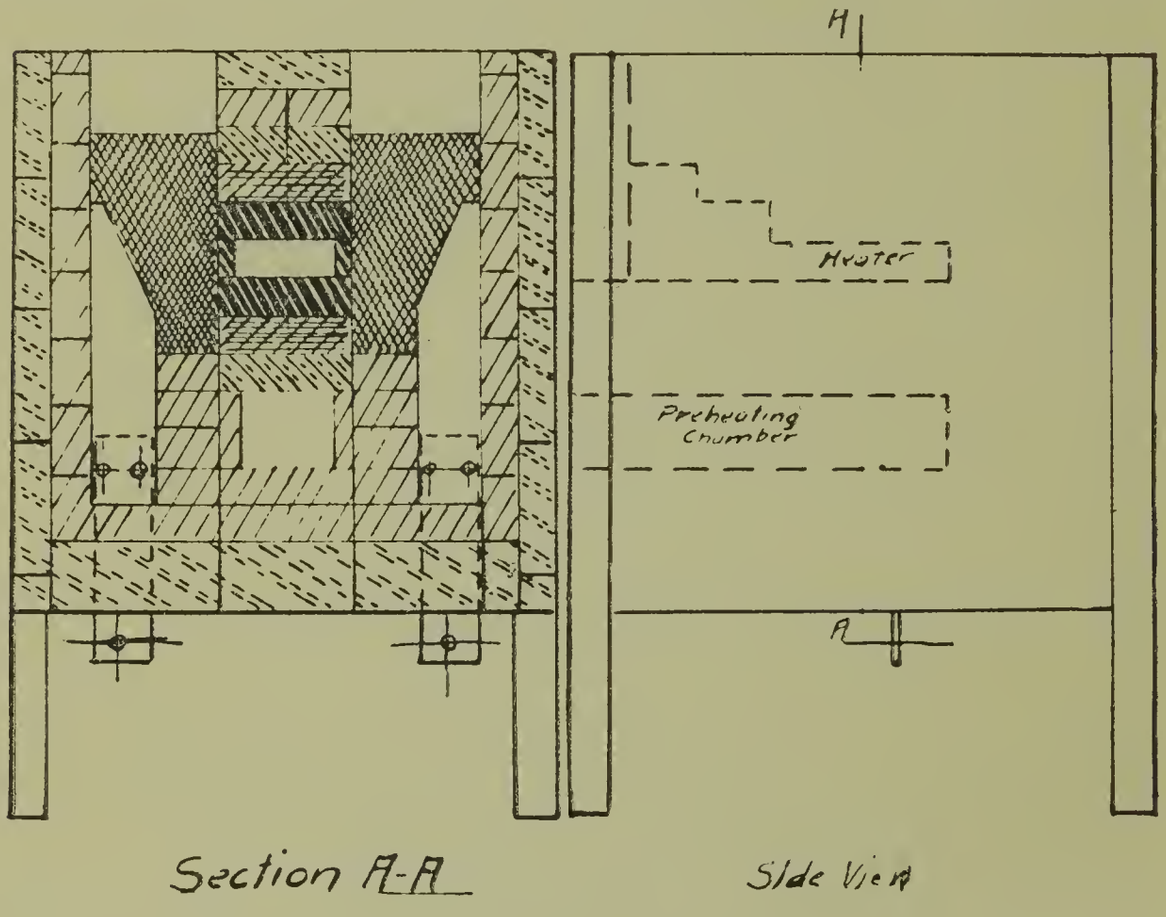


FIG. 6.

efficient which the material was known to possess. This, however, has not proven a serious handicap. In fact, it is possible to use this negative temperature coefficient characteristic as the basis for the control.

A furnace was built up using this material as a resistor, but arranged to heat from the roof as well as from the hearth. A small size was purposely chosen in order to facilitate the construction work and testing. The general design of this furnace is shown in the accompanying illustrations (Figs. 6 and 7). The resistor was 2½ in. (6.3 cm.) thick and the surface of the hearth

was covered with approximately ⅛ in. (0.32 cm.) alundum cement, for mechanical protection. The surface of the roof heater was not protected in any way. It has been possible to obtain continuous operation at temperatures as high as 1,250° C., without injury to any part of this furnace. The temperature drop through the silicon carbide was a great deal less than that which had been observed through the granulated resistance bed, and the danger of damaging the lining was very much reduced.

On account of the great negative temperature coefficient of

FIG. 7.

resistance, it is necessary to provide special arrangements for voltage control of this type of furnace. The accompanying curves show quite distinctly the variations in furnace resistance. Fig. 8 shows the starting characteristics as the furnace is heated up from cold. A large number of voltage steps were available on the supply transformer used for the experiments, although it is not necessary to use more than three or four steps in regular practice. It can be seen that the current rose gradually during the first 40 minutes until 350 amperes was reached, at which point the voltage was reduced to 43.5 volts. From this point the voltage was gradually

reduced in small steps down to 25 volts at the end of two hours and 40 minutes. At this time the furnace had reached a temperature of approximately 1,100° C., and was ready for operation. It was permitted to remain on 25 volts over night and the next morning the current had dropped to approximately 250 amperes. Fig. 9 shows the second test performance when the furnace was used for heating steel tools for forging. This work was carried on for a period of two hours in the morning and three hours in

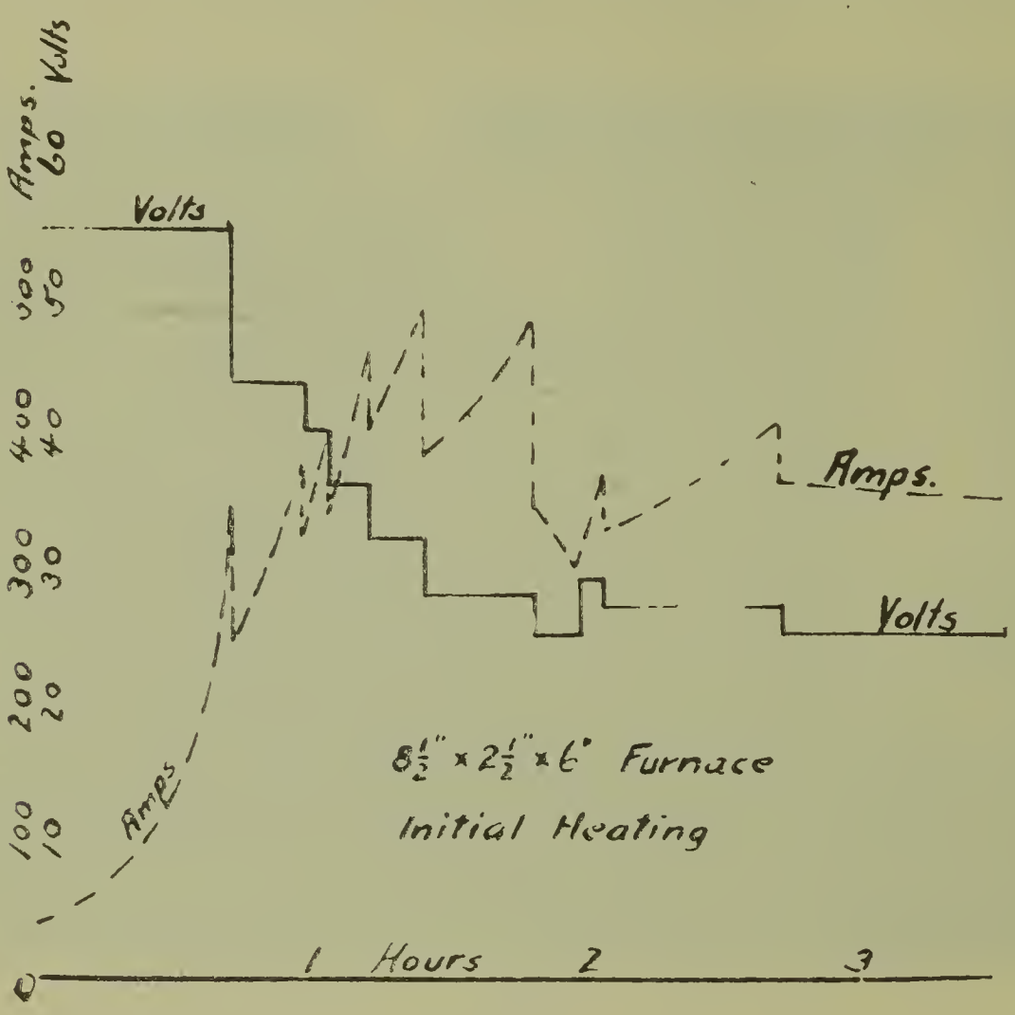


Fig. 8.

the afternoon. It will be noticed that very little change was needed in the voltage to maintain working conditions. This particular operation was in the nature of a preliminary test and no effort was made to speed up production.

From the preceding curves it can be seen that an operating voltage range of plus and minus 20 percent from the average should be sufficient to take care of all conditions. This should be available in several steps. The apparatus should operate automatically, and should maintain approximately a constant current.

In this way approximately constant temperature will be maintained, as the resistance of the furnace itself is serving as the operating element of the thermostat.

This regulation can also be obtained by using a voltage regulator operated by a constant current relay. It is probable that this arrangement would maintain the current more nearly constant, but it would be slightly more expensive and would probably not present any decided advantages over a step-by-step control.

Starting voltage must be from two to three times the normal operating voltage. The circuit should be provided with current-limiting relays or circuit breakers, so that the furnace current will not exceed the safe maximum at any time. This is a safeguard against excessively rapid heating up from cold. If the initial

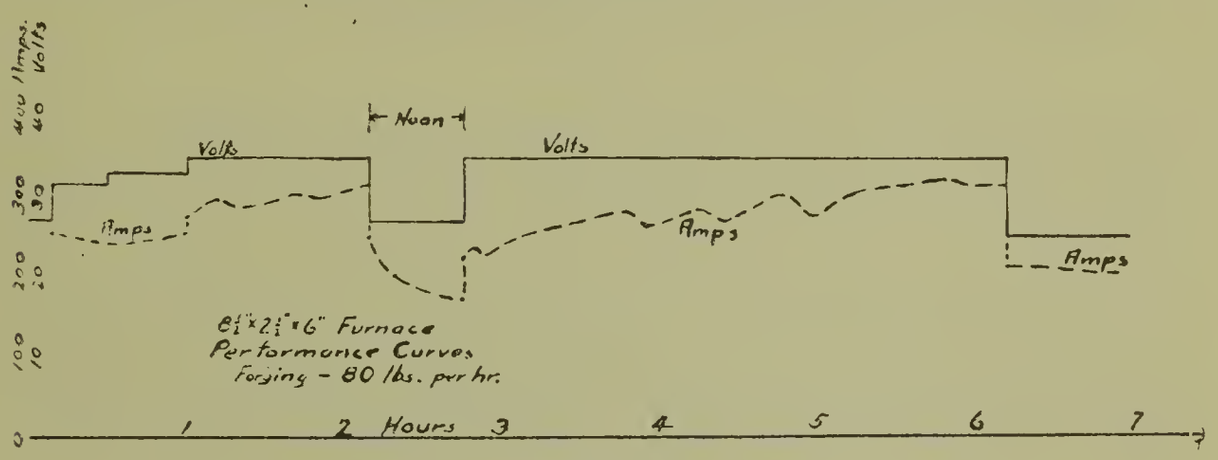


FIG. 9.

heating takes place at too high a rate, there is danger of concentration of current through the centers of the resistors which might carry the temperature up so rapidly as to injure the material before the temperature on the surfaces and in the furnace chamber had come up to the proper value.

A further test of this construction has been carried out in the large furnace first mentioned. The granular resistance bed has been removed and replaced by one built up of silicon carbide bricks (Fig. 10). This furnace has been in use for experimental purposes for several months and has been subjected to a large number of heating runs under severe conditions. Temperatures up to 1,200° C. have been obtained without difficulty, although the distribution of the heat is not suitable for commercial heating for forging, or similar operations. This furnace has been used with success for hardening tools and dies, and for firing porcelain.

The lack of combustion of the resistor material results in there being an oxidizing atmosphere within the furnace. A non-oxidizing atmosphere has been obtained quite successfully, however, by spreading granulated charcoal or carbon over the surface of the resistor, near the door, where it was burned by the air which entered.

It is realized that there are many problems in connection with the successful commercial application of this type of electric

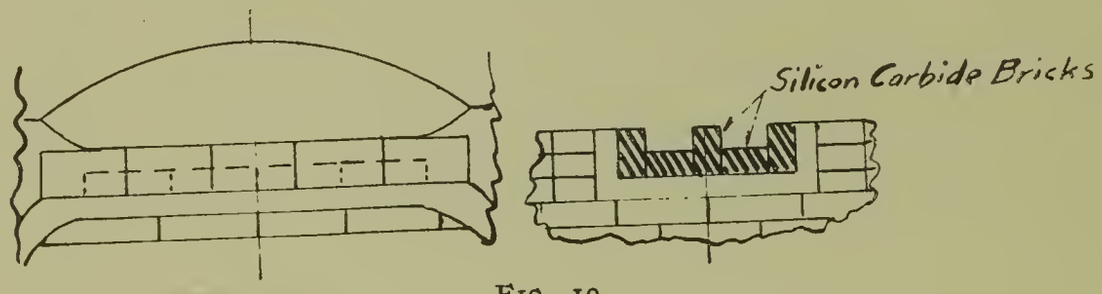


FIG. 10.

furnace which have not been considered in this discussion. Practically every commercial industrial heating application requires special study in order to insure most economical operation. Electricity can be supplied most economically if the generating and distributing equipment is loaded constantly to its full capacity, and electric resistance furnaces will be most satisfactory forms of load if operated continuously. Repeated heating and cooling is likely to cause a more rapid depreciation of any furnace than continuous operation, and it is therefore to be expected that this type of furnace should be most suitable for applications which are constant and continuous, and not intermittent.

DISCUSSION.

PRESIDENT C. G. FINK[1]: How does silicon carbide compare with metallic silicon as a resistor?

F. THORNTON, JR.: I cannot answer that question, as I have not had any experience with metallic silicon.

F. A. J. FITZGERALD[2]: If the question is not indiscreet: Is the silicon-carbide brick used in the furnace made with some binding material, or is it simply recrystallized silicon carbide?

[1] Director of Research, Chile Exploration Co., New York City.
[2] FitzGerald Laboratories, Niagara Falls, N. Y.

Another question: After long use, has any trouble been experienced due to a change in the resistance of the silicon-carbide brick, a permanent change due to the vaporization of the silicon and the consequent formation of graphite?

F. THORNTON, JR.: Mr. Chairman, as I understand it, the material used was a pure silicon carbide, as pure as was obtainable. There was no binder in it, except the slight impurities which might have been in the silicon carbide.

As to the second question: The performance of the material seems to be limited by the temperature of the operation. Furnace temperatures up to 1,200° C. do not affect the material. But if the temperature is carried beyond that, after a short time, it soon shows up in the action mentioned.

HORACE W. GILLETT[3]: To me this is an extremely exasperating paper.

Fig. 6 has no legend, and you cannot tell what is what. In general the experiments are not described so they could be duplicated by another worker.

I wish instead of giving a mere outline he would tell more of his failures. I want to know, and to have on record, what Mr. Thornton knows; but I cannot get it from this paper. I request that Mr. Thornton be asked to rewrite this paper so as to give us some real information.

I should like to know whether he had cement between his brick, or whether they were butt joints, and how he started with 55 volts. I always understood it took a high voltage to start a cold carborundum resistor, and it would be useful to know the conditions for starting at low voltage.

F. THORNTON, JR.: It is a little hard to answer these questions. There are many things about experimental failures sometimes that one does not understand and cannot briefly explain. I realize that this paper does not go thoroughly into detail, and in fact, there are a number of details which, if they had been included, would have extended the discussion interminably.

As for the details regarding power consumption, watts per square inch, and rate of production, those are still matters of investigation, and if any statements were made they would be

[3] U. S. Bureau of Mines, Ithaca, N. Y.

open to further revision later on. Let us hope that at a later day the information mentioned as desirable by Mr. Gillett can be given out authoritatively, and with some assurance that it will not have to be corrected. The voltage for initial heating is a matter of time. It is true that carborundum has a high negative temperature coefficient, so that to start heating takes a long time unless an excessively high voltage is used; and if such is used, it is liable to cause concentration of the heat, causing localized rises of temperature, and creating an over-heating, and the effect mentioned by Mr. FitzGerald might be started. Therefore, it is necessary to slow down the initial heating of any mass of carborundum operating at the voltage of the furnace described.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

RESISTANCE TYPE FURNACE FOR MELTING BRASS.

By T. F. Baily.*

[Abstract.]

Running tests are quoted of an electric resistance furnace melting bronze and brass, with mechanical tests of the cartridge brass produced and numerous analyses showing the uniformity of the metal at the beginning and end of the cast. The electrical power required shows favorable comparison of melting costs with crucible or open-flame melting furnaces, the average being 450 K.W.H. per short ton of bearing bronze (495 per metric ton), and 311 K.W.H. per short ton of cartridge brass (342 per metric ton).

The furnace described in this paper is of the non-crucible type and operates on the true resistance principle. A general description of the furnace is as follows:

In a cylindrical shell, supported by the usual trunnions and brackets for tilting, is constructed suitable walls and roof forming the furnace chamber. The bottom of this chamber is so shaped as to make a bowl-shaped hearth for containing the metal when melted.

The resistance element is contained in a circular trough made of a refractory material similar to carborundum fire sand. In this refractory trough the resistance material, composed of broken carbon, is placed, forming contact with the electrodes which are placed diametrically opposite each other with their ends protruding into the circular trough.

The control of the current, and hence of the furnace temperature and melting capacity, is by means of suitable voltage taps brought out from the secondary of a special transformer supplied with each furnace.

* President, The Electric Furnace Co. of America, Cleveland, Ohio.

TABLE I.

Operating Sheet.

105 kw. Tilting Type Furnace Making Brass Cartridge Case Slabs.
Week of August 6, 1917.

Charge No.	Pounds Charged	Composition of Brass	Percent Loss Chem. Analysis	Percent Loss by Weight	Average Capacity per hour	Time to Melt Charge to Ch'ge hrs. min.	Time Lost in Pouring min.	K.W.H. Charge to Ch'ge	K.W.H. per Ton
1	750.0	66–33%	...	...	257	2 – 55	20	253	675
2	757.5	" "	2.97	...	325	2 – 20	35	152.6	403
3	757.0	" "	0.66	...	325	2 – 10	20	183.6	485
Day Av.	754.8	.....	...	...	305	2 – 28	25	196.4	520
4	753.0	66–33%	0.00	...	329	2 – 17	18	138.9	379
5	759.0	" "	0.80	...	407	1 – 52	17	144.1	380
6	775.0	" "	0.64	...	452	1 – 45	15	157.3	406
7	756.0	" "	0.79	...	378	2 – 0	25	147.7	390
8	776.0	" "	1.03	...	423	1 – 5	20	145.4	375
Day Av.	763.8	.....	0.85	...	394	1 – 56	19	146.7	385
9	725.0	69–31%	0.55	...	363	2 – 0	12	173.1	477
10	752.0	68–32%	0.13	...	410	1 – 50	13	151.8	403
11	748.0	" "	0.13	...	360	2 – 5	15	172.6	460
12	730.0	" "	0.00	...	437	1 – 40	13	143.7	394
13	732.0	" "	0.27	...	462	1 – 35	20	103.0	282
Day Av.	737.4	.....	0.22	...	402	1 – 50	14.6	148.4	402
14	732.0	68–32%	1.91	0.95	374	1 – 58	12	154.1	422
15	733.0	" "	2.04	1.37	449	1 – 38	13	143.0	390
16	747.0	" "	0.67	0.94	560	1 – 20	12	116.6	312
17	740.0	" "	1.08	1.22	517	1 – 26	12	96.4	261
18	750.0	" "	2.67	1.08	562	1 – 20	10	99.8	267
19	755.0	" "	1.72	1.99	628	1 – 15	11	86.5	229
Day Av.	744.5	.....	1.68	1.25	503	1 – 30	11.6	116.0	312
*20	766.0	68–32%	...	0.78	548	1 – 24	10	118.8	310
*21	766.0	" "	...	2.48	638	1 – 12	10	108.7	284
22	740.5	" "	...	4.93	705	1 – 3	10	76.8	208
23	773.5	" "	...	2.13	483	1 – 36	10	106.2	275
24	765.5	" "	...	2.16	567	1 – 21	11	100.9	264
25	766.5	" "	...	3.45	567	1 – 21	15	96.0	251
26	766.5	" "	...	2.55	576	1 – 25	10	114.7	299
Day Av.	763.5	.....	...	2.64	575	1 – 20	11	103.1	270
27	758.5	68–32%	...	5.40	523	1 – 27	14	109.0	287
*28	769.5	" "	...	0.98	607	1 – 16	11	122.6	319
*29	771.0	" "	...	1.55	526	1 – 28	13	111.5	289
*30	768.0	" "	...	1.69	650	1 – 11	12	87.9	229
*31	766.0	" "	...	2.22	638	1 – 12	12	102.1	267
*32	763.0	" "	...	1.51	587	1 – 23	13	90.7	236
Day Av.	766.0	.....	...	2.22	584	1 – 19	12.5	104.0	272
Week Av.	752.0	.....	...	1.52	488	1 – 40	14.5	128.4	340

Charges 1 to 19 inclusive were new metal, copper and zinc ingots.

Charges 20 to 32 inclusive were 50 percent new metal, copper and zinc ingots, and 50 percent scrap brass, which contained about 8 pounds per charge of material not metal. A total of 46.5 pounds of this material was removed from furnace.

Loss average for all charges of 50 percent scrap was 2.44 percent gross, or eliminating 46.5 pounds was 1.97 percent net, or allowing 8 pounds material not metal per charge was 1.39 percent net.

* In these charges the zinc was added after copper and scrap had melted; all the remaining charges composed of 50 percent scrap—50 percent new metal entire charge was added at one time.

Loss average for all charges marked * was 1.60 percent gross, or allowing 8 pounds material not metal per charge was 0.56 percent net.

Metal was poured from furnace to ladle and from ladle to mold in all cases.

The rated electrical capacity is 105 K.W. for a furnace shell 7 feet (2.1 m.) in diameter, having a hearth capacity of 1,500 pounds (680 kg.) and an average melting rate of 600 pounds (270 kg.) per hour.

Table I is a tabulation of a typical week's run on a 105 K.W. furnace melting ingot and scrap material for the manufacture of yellow brass slabs 6 x 1¼ x 42 inches (15 x 3.2 x 107 cm.). This material is used for the manufacture of cartridge case discs for 3- and 4-inch (7.5 and 10 cm.) shells.

It is to be noted that for the six days of this period the average figures for the 32 heats were as follows:

Average number of working hours per day.....8 hr. 45 min.
Average number of heats per day.....5.33
Average weight of heats, each.....752 lb. (342 kg.)
Composition of metal.....68 Cu.-32 Zn
Average capacity per hour.....488 lb. (222 kg.)
Average time per heat.....1 hr. 40 min.
Average time lost in pouring.....14.5 min.
Average kw. hours per heat.....128.4 kw. hours.
Average kw. hours per ton (2,000 lb.).....340 kw. hours.
Average kw. hours per metric ton.....374 kw. hours.

Table II.

Physical Properties of Electrically Melted Brass.

Lot No. 1: Copper, 2; Zinc, 1.

No.	Dimensions Inches	Area Sq. In.	Breaking Point Lb.	Tensile Strength		Elongation Percent in 2 in. (5 cm.)
				Lb./sq.in.	Kg./sq.mm.	
A	0.991 x 0.367	0.36374	17,355	47,713	33.4	64.5
B	0.992 x 0.369	0.36595	18,965	51,825	36.1	56.5
C	0.979 x 0.369	0.36125	18,030	49,910	35.0	60.0

Lot No. 2: Copper, 69.0; Zinc, 31.0.

1	0.984 x 0.350	0.34440	15,700	45,545	31.9	67.5
2	0.986 x 0.347	0.34214	15,350	44,865	31.4	71.5
3	0.989 x 0.352	0.34813	16,445	47,235	33.0	64.0

The physical properties of metal cast in the above runs from test bars taken at random were as shown in Table II.

To determine what variations there might be between the first and last bar cast from each heat, the analyses shown in Table III were made covering heats 1 to 32.

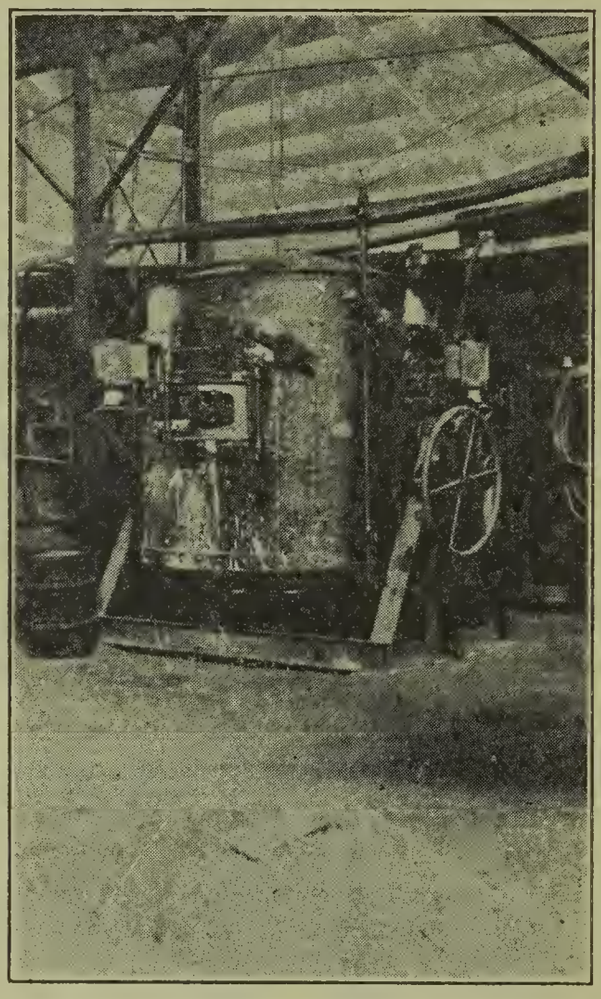


Fig. 1.

These runs were all made in the furnace shown in Fig. 1.

Fig. 2 illustrates a furnace of the same type just described used in the manufacture of copper and zinc alloys.

Table IV shows the operating characteristics of one of these furnaces when operating on 400-lb. heats of "red brass." Each charge consisted of 352 lb. (160 kg.) copper, 40 lb. (18 kg.) tin, and 8 lb. (3.65. kg) phosphorus.

It is to be noted that the average time per heat was 1 hour 4 minutes, indicated K.W. 86; K.W.H. per heat 90.4; K.W.H. per short ton 450.3, per metric ton 495.

TABLE III.

Analyses taken on the first bars cast from the heat and the last bars cast from the same heat. Heats 1 to 32 inclusive.

No.	Percent of Copper		No.	Percent of Copper	
	First Bar	Last Bar		First Bar	Last Bar
1	....	....	17	68.37	69.72
2	68.70	....	18	69.34	70.02
3	66.73	67.64	19	68.67	70.71
4	65.97	66.73	20	68.97	70.17
5	66.42	67.33	21	68.73	69.80
6	66.43	67.18	22	69.70	70.77
7	67.20	67.36	23	69.10	69.10
8	66.90	67.05	24	68.94	69.55
9	68.42	70.25	25	68.94	70.46
10	67.66	69.34	26	69.70	70.46
11	67.59	69.03	27	73.28	74.12
12	68.42	68.88	28	69.70	70.31
13	68.73	68.73	29	69.25	69.85
14	69.65	69.87	30	69.40	69.55
15	69.34	70.78	31	68.94	70.00
16	68.52	69.42	32	69.55	69.55

TABLE IV.

Tests on Melting Furnace at Lumen Bearing Company, Buffalo, N. Y., June 1, 1917. (Charges 400 lb. = 182 kg.).

Heat	Metal Charged		Time of Heating	Av. Ind. K.W.	K.W.H. Consumed	K.W.H.	
	Kind	Weight lb.	hr. min.			Per 2,000 lb.	Per 1,000 kg.
1	Cu.....353 Sn..... 40 P..... 8	401	1 – 17	78	99.7	496	546
2	Cu.....352 Sn..... 40 P..... 8	400	1 – 10	87	101.1	500	550
3	Cu.....352 Sn..... 40 P..... 8	400	.. – 55	84	77.5	386	425
4	Cu.....352 Sn..... 40 P..... 8	400	.. – 55	102	93.8	470	517
5	Cu.....352 Sn..... 40 P..... 8	400	1 – 5	76	82.1	410	451
6	Cu.....352 Sn..... 40 P..... 8	400	1 – ..	88.2	88.2	440	484
Average.....			1 – 4	86	90.4	450	495

One of the same furnaces making yellow brass of the composition of 72 percent copper and 28 percent zinc from ingot, oper-

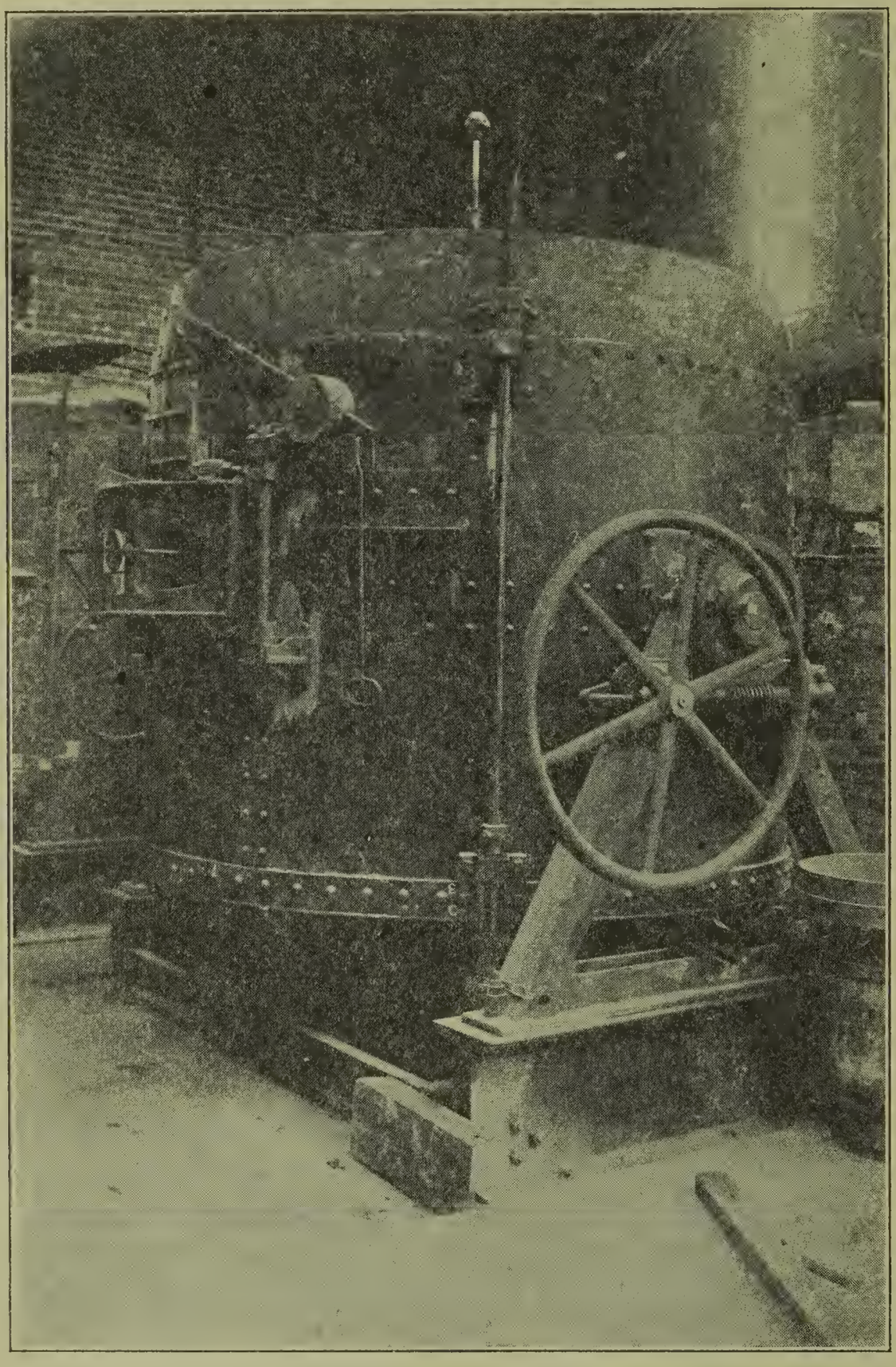


FIG. 2.

ating with 816-lb. (371 kg.) heats, shows the results as given in Table V.

Table V.

Yellow Brass-Metal From Ingot. June 8-9, 1915.
(Charges 816 lb. = 311 kg.)

Heat	Metal Charged Percent	Time of Heating hr.	min.	Av. Ind. K.W.	K.W.H. Consumed	K.W.H. Per 2,000 lb.	K.W.H. Per 1,000 kg.
3	Copper..72 Zinc.....28	1	32	105	159.55	395	435
4	Copper..72 Zinc.....28	1	15	103	129	313	344
5	Copper..72 Zinc.....28	1	12	118	142	350	385
1	Copper..70 Zinc.....30	1	12	84	101	247	272
2	Copper..70 Zinc.....30	1	19	79	104.7	256	282
3	Copper..70 Zinc.....30	1	20	93	124.6	306	337
Average.....		1	18	97	126.8	311	342

It is to be noted that the average time of heat was 1 hour 18 minutes, indicated K.W. 97; K.W.H. per heat 126.8; K.W.H. per short ton 311, per metric ton 342.

The first furnace of the type described in this paper for melting alloys was put into service in August, 1916, and was used exclusively for the production of a special bearing metal, having the trade name of "Lumen" and containing a high percentage of zinc. A photograph of this furnace is shown in Fig. 3.

The second furnace of the type was installed at the same plant over six months after the first furnace was installed, and has been used for the production of brass and manganese castings, as well as for the bearing metal mentioned.

Fig. 4 shows both of these furnaces in operation, one of them pouring yellow brass, the other pouring Lumen metal.

The economic value of these furnaces for brass melting must be readily apparent to anyone comparing costs calculated from the figures given, with the present cost of melting in pit-type crucible furnaces, or the cost in open-flame furnaces; and though the brass industry has long been skeptical of all electric brass

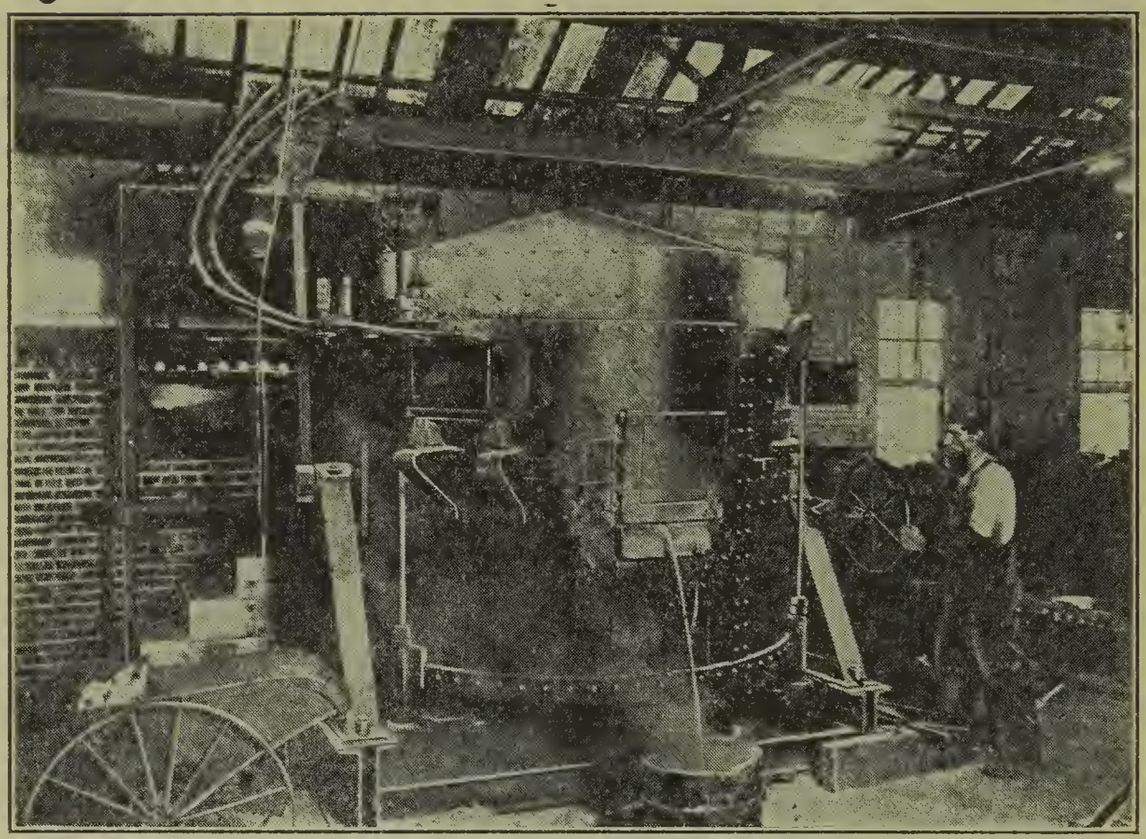


FIG. 3.

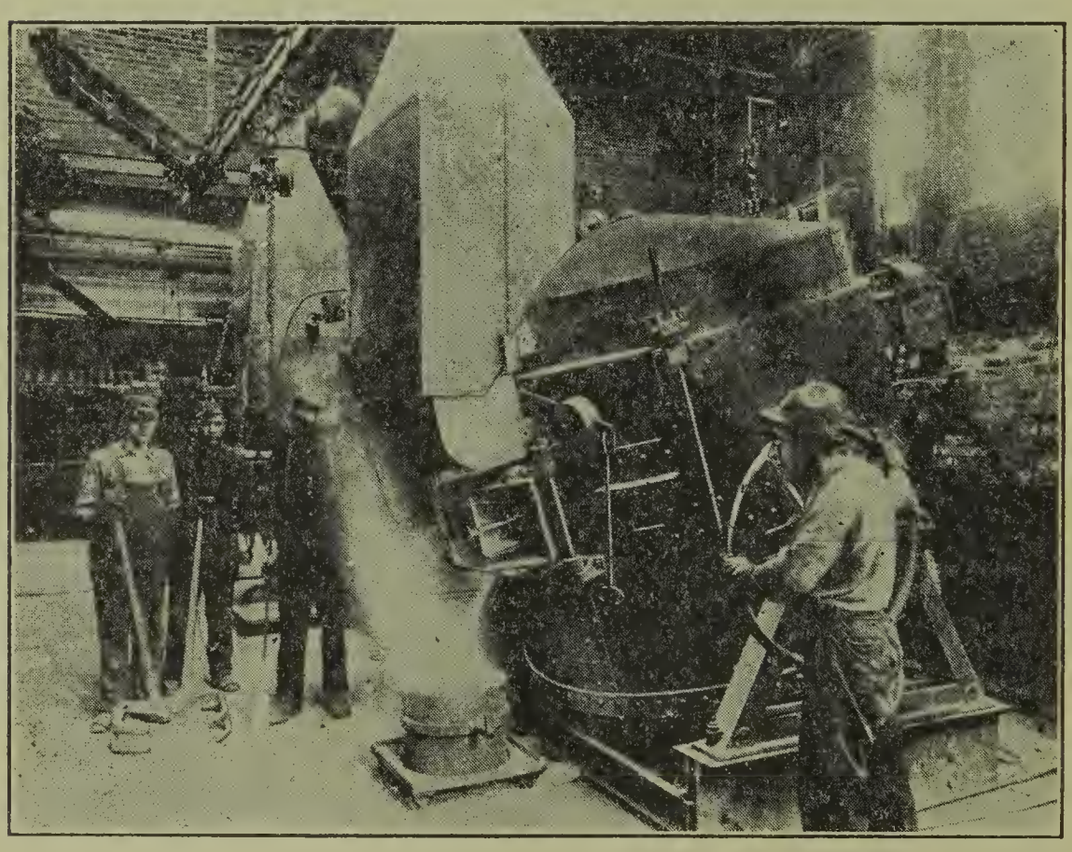


FIG. 4.

furnaces, the work actually accomplished by the type of furnace described, in commercial practice, has fairly entitled it to the favorable reception it is now receiving.

DISCUSSION.

T. F. Baily: Zinc running 0.80 to 15 percent copper has been produced with three renewals of troughs in that time, no renewals in the way of roofs or brick. In one of the other furnaces in which the runs were made, one furnace has made about 150 tons of yellow brass. It has never operated on anything else.

H. W. Gillett[1]: Mr. Baily evidently thinks in terms of 24-hour operation. In one way he has over-estimated his results, and in the other way he has under-estimated them. In other words, his average here in the abstract needs some explanation or some illustration which is not given.

I had no opportunity to talk with the people operating this furnace, and this Table I does not show that the furnace is heated at night. About one-third of the day power input is used on it at night, and if the brass foundryman reads this and does not know it was kept hot at night, he might think he can do the work at the stated power consumption without night heating. As a matter of fact, taking into consideration the night operation, the figures will be nearly doubled.

On the other hand, on the 24-hour basis, he could get down to 275 kilowatt-hours per ton. That is not thoroughly clear, and I think it should be made clear in order that the foundryman may know just what is happening.

In Table I, I am glad to see the percent losses by chemical analysis and percent losses by weight are both given.

If one takes a ladle and dips out half his bath, then analyzes the remaining metal, the analysis will not show that anything has been taken out. Similarly, if there is a loss by oxidation, say, of all the constituents of an alloy, analysis will not give true loss

[1] U. S. Bureau of Mines, Ithaca, N. Y.

figures, for that method assumes that some one constituent, taken as a base, suffers no loss. Hence taking losses by actual weight of metal in and metal out, is the better method.

The segregation figures given would not be considered very good practice by most brass men, and indicate that thorough rabbling is needed in this type of furnace to give a well-mixed alloy.

T. F. BAILY: We are very glad not to let any foundryman have any impression that is erroneous, any misapprehension of what we are doing.

J. W. RICHARDS[2]: Mr. Baily kindly gave me a chart of seven heats of brass run in a ten-hour day, and I figured out the electrothermal efficiency as 64 percent.

We have two figures, one from the Bureau of Standards and the other from Mr. Bennett, as to the amount of heat in melted brass. We note that at 1,100° C., using 90 kilowatts, the efficiency figures 64 percent. If the furnace used 120 kilowatts, the efficiency would be 74 percent. That would mean that you could run ten heats in a 24-hour day instead of seven; which points to the advantage of working the furnace as hard as it can stand being run.

[2] Professor of Metallurgy, Lehigh University.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

PREPARATION OF PURE ALLOYS FOR MAGNETIC PURPOSES

By Trygve D. Yensen.*

[Abstract.]

The author discusses the reasons why pure iron, and iron containing elements which do not separate its crystals from each other, have great magnetic permeability and low hysteresis losses. He reviews the literature of this subject. He describes an electrolytic plant for producing pure iron, and the various commercial forms in which the alloying elements to be used are obtained. Detailed description is given of electrically fusing magnesia, and moulding and baking crucibles out of the product. Ordinary and vacuum electric furnaces for making the alloys, at temperatures up to 1,800° C., are described; also annealing furnaces for heat treatment of the alloys produced. An illustration of the magnetic testing laboratory is shown, with catalogue of its outfit.

I. General Considerations.

In spite of the important improvements made during the last decade in the magnetic quality of iron and iron alloys used in commercial machinery, there is still room for further improvements. This became especially evident in the work done at the Engineering Experiment Station of the University of Illinois during the last five years, in which work the author was actively engaged until last year.[1]

* Research Metallurgist, Westinghouse Elect. & Mfg. Co.

[1] Bulletins Nos. 72, 77, 83, 95, Eng. Exp. Sta., Univ. of Ill.
Trans. Am. Inst. Elect. Engrs., 33, I, 451 (1914); 34, 2455 (1915).
Proc. Am. Inst. Min. Engrs., Feb., 1916.
Gen. Elect. Rev., 18, 881 (Sept., 1915).

This investigation has shown that it is possible to obtain iron and iron alloys with magnetic permeabilities as high as 50,000, as compared with 6,000 to 8,000 in the best grade of commercial material, and with a hysteresis energy loss as low as 600 ergs per cm. cube per cycle for $B_{max} = 15{,}000$ as compared with 3,000 to 4,000 for commercial silicon steel.

The attainment of these results was made possible by means of very pure materials. Those who are familiar with the characteristics of magnetic circuits know very well what a tremendous effect is produced in the magnetic flux of a solid iron ring by making a cut across it and afterwards joining the two ends. Even if the ends are well ground so that they butt up against each other without any appreciable gap, the magnetic flux of the ring will be very much smaller than it was before the ring was cut. This applies particularly to the case where the ring is magnetized to some point on the steep part of the magnetization curve. The same consideration may be applied to the internal structure of the iron. This structure is chiefly made up of iron crystals or grains of various sizes and forms. In the case of pure iron, these crystals come very close together. If now certain foreign elements are dissolved in the molten iron, some of these will, as the iron cools, remain in solution (solid solution) even after the iron has cooled down to room temperature, while others will be precipitated along the road at different temperatures. These foreign elements that are thus thrown out of solution will occupy the spaces between the iron crystals and leave them further apart than would be the case without these impurities. Furthermore, a number of elements like carbon, sulphur, phosphorus and oxygen, are not precipitated as such, but rather in various combinations with iron. Thus carbon is precipitated as Fe_3C, called cementite, with magnetic permeability much lower than that of pure iron and with much higher hysteresis loss. Calculating the percentage of cementite from the percentage of carbon present, the ratio is 15 to 1. If, for instance, the iron contains 0.1 percent carbon, there will result a precipitate between the pure iron crystals of 1.5 percent (by weight) of cementite.

From this consideration, it is quite evident that even very small amounts of carbon will have a marked effect upon the magnetic properties of pure iron. In a similar way sulphur will be precipitated as FeS, forming a web along the grain boundaries, or if manganese is present sulphur will be precipitated in less harmful form as MnS globules. Phosphorus is precipitated as Fe_3P if carbon is present in appreciable quantities, otherwise it may go into solid solution with the iron. Gases like hydrogen, nitrogen, and oxygen, are readily dissolved by molten iron, but are given off as the iron solidifies, forming cavities between the Fe crystals, the effect of which may be even worse than that of carbon, sulphur or phosphorus.

It was stated above that certain elements remain in solid solution with the iron as the latter cools. Such elements, *e. g.,* silicon or aluminum, have generally much less effect upon the magnetic properties than have impurities like carbon that are precipitated between the iron crystals. They may even have a beneficial effect by precipitating some of the harmful impurities in a less harmful form. Silicon, for instance, when present in sufficient quantity will precipitate carbon as graphite instead of cementite.

It is very fortunate that these elements can be introduced into pure iron without destroying its magnetic properties, for they serve a very useful purpose in increasing the electrical resistance of the iron. This is of great importance in connection with the eddy current loss in electrical machinery, for the greater the resistance of the iron the less is the loss. Four percent silicon added to the iron will increase the electrical resistance from 10 microhms per cm. cube to over 50 microhms, and this is accomplished without any material change in the magnetic permeability at low and medium flux densities.

At high flux densities, however, all elements, outside of nickel and cobalt, act like impurities in iron, in that they lower its permeability in direct proportion to the amount present. In other words, they simply act like diluents, diminishing the capacity of the iron for carrying the magnetic flux. Thus 4 percent silicon lowers the flux density by over 10 percent, and 0.1 percent carbon

lowers it by 4 to 5 percent. It is therefore of the utmost importance whenever high flux densities are required to use iron of the highest purity.

Until recently it was generally believed that no material had a higher magnetic saturation value than pure iron.[2] In 1912, however, Weiss[3] discovered, and his discoveries have since been amply verified,[4] that when cobalt is added to iron the saturation value is markedly increased, and that the increase is in direct proportion to the amount of cobalt added, up to 34.5 percent, corresponding to the compound Fe_2Co. This compound, if free from impurities, has a saturation value 10 to 13 percent higher than that of pure iron. This is the more interesting because cobalt by itself has a much lower saturation value than has iron. The increase, therefore indicates a change in the molecular arrangement of the crystal. The discovery of the properties of the iron-cobalt alloys opens up an entirely new field of magnetic research.

Nickel[5] acts quite oppositely to cobalt, except that certain investigations[6] tend to show that small amounts of nickel (up to 5 percent) increase the permeability of iron at high flux densities. This point, however, needs further confirmation before it can be accepted, as the data on the subject is very meagre. Furthermore, impurities play such a large part in the results obtained that the greatest care must be exercised in drawing conclusions.

It is believed that sufficient has been said to show the importance of impurities in iron for magnetic purposes. The above considerations may be summarized as follows:

1. The magnetic properties at low and medium flux densities depend very largely upon the crystal boundary conditions. With no impurities present such as will be precipitated between the crystals, it is possible to obtain iron and iron alloys with exceedingly high permeabilities and with correspondingly low hysteresis losses.

[2] Hadfield & Hopkinson, Inst. Elect. Engrs., **46**, 225 (1911).
Gumlich, Trans. Faraday Soc., **8**, 109 (1912).

[3] Comptes Rendus, **156**, 1970 (1913); Trans. Faraday Soc., **8**, 149 (1912).

[4] Yensen, Gen. Elect. Rev. *Loc. cit.*

[5] Weiss. *Loc. cit.*

[6] Burgess and Aston, Metal. and Chem. Engr., **8**, 23 (1910).

2. The electrical resistance of iron can be enormously increased without affecting the magnetic properties at low flux densities, by the addition of certain elements that form solid solutions with the iron.

3. All addition elements thus far investigated except cobalt, and possibly nickel, whether they are precipitated between the iron crystals or form solid solutions with the iron, lower its magnetic saturation value.

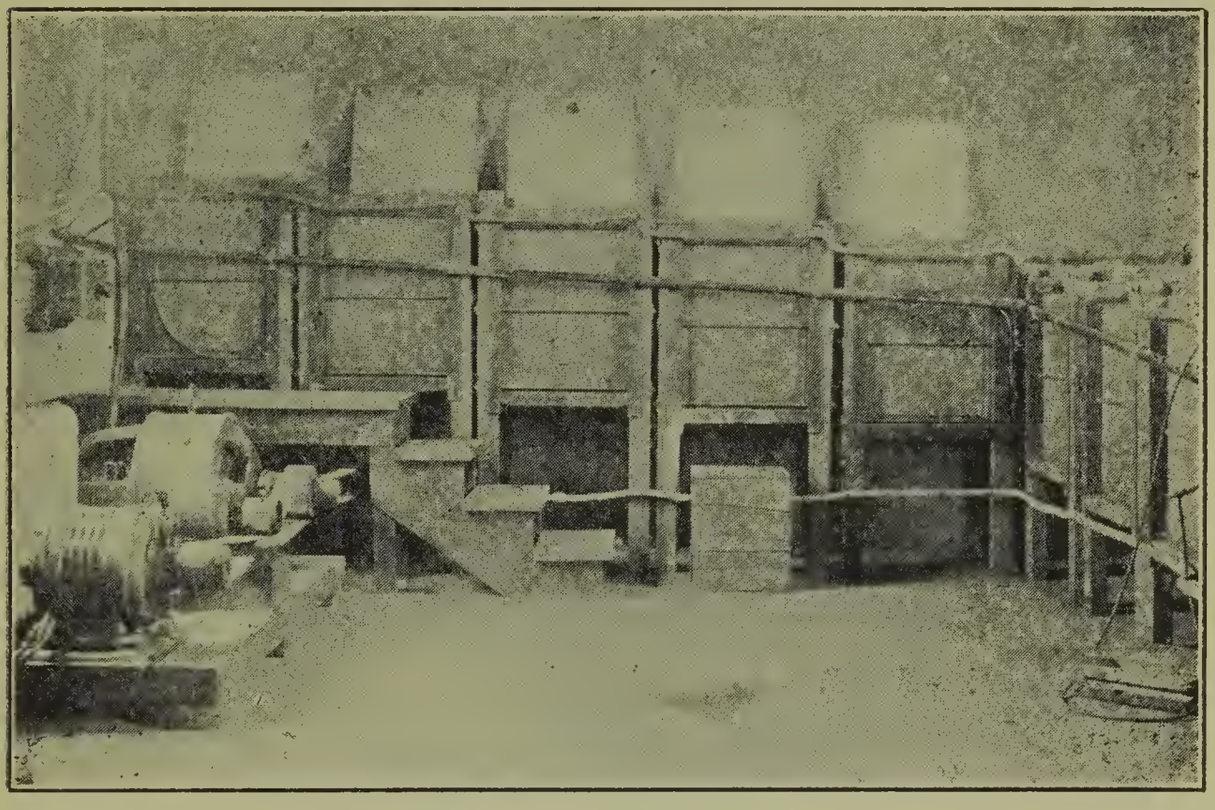


FIG. 1. Electrolytic Iron Refining Plant. Westinghouse Research Laboratory.

4. Cobalt up to 34.5 percent increases the magnetic saturation value of iron in proportion to the amount added, but these iron-cobalt alloys are just as susceptible to impurities as is pure iron.

II. ELEMENTS USED.

The first step in the preparation of pure iron and pure iron alloys is necessarily the securing of pure elements to start with. Below will be found a list of some of the more important elements needed in investigations of this kind, their commercial forms and their chemical analysis.

Iron.

Electrolytic iron is the most reliable source of iron. It is not on the market and must generally be prepared by the investigator himself. The methods have been thoroughly worked out by C. F. Burgess and his associates at the University of Wisconsin,[7] and can be modified to suit local conditions. A photograph of the plant at the Westinghouse Research Laboratory is shown in Fig. 1.

The electrolyte consists of 250 g. ferrous sulphate, 150 g. ferrous chloride and 200 g. ammonium sulphate.

The anodes consist of the purest commercial iron obtainable, *viz.*, "American Ingot Iron." The iron is first deposited on lead plates, and when the deposit is about ½ inch (1.25 cm.) thick, these are transferred to another set of tanks and used as anodes for a second refining. In the second set aluminum cathodes are used, from which the iron can be readily stripped provided the aluminum plates are kept in good condition. This is done as follows:

1. Remove traces of Fe and Fe_2O_3 after the deposit has been stripped off, by means of 1 : 1 HNO_3.
2. Wash plates and neutralize the acid by means of $NaHCO_3$.
3. Wash with H_2O.
4. Straighten out plates.
5. Remove scratches by polishing with fine emery.
6. Remove Al_2O_3 by polishing and buffing.
7. Treat with alcohol and ether, and finally dry the plates.

This treatment will leave the plates with a thin film of aluminum oxide to which the iron will not adhere to any extent.

The current density is about 1 ampere per sq. dm. of electrode surface. The electrolyte is circulated through the tanks by means of a small hard-lead centrifugal pump, that lifts the electrolyte from the lower tank up to the top tank from which it flows through the other tanks by gravity. The total energy consumption is about 1 kw. hour per kilogram of iron deposited.

[7] Burgess and Hambuechen, Iron and Steel Mag., 8, 48 (1904).
Watts and Li, Trans. Am. Electrochem. Soc., 25, 529 (1914).

After being stripped off the aluminum anodes, the iron is well rinsed with hot water to remove most of the electrolyte and is then stored away. Before being used, however, it is put through a thorough cleaning by means of successive baths of dilute HCl, boiling distilled water, alcohol and ether, after which it is dried in vacuo.

Analysis of a sample of iron thus made is as follows:

S	0.003	percent
P	0.003	"
C	0.010	"
Mn	Trace	"
Si	0.004	"
Cu	Trace	"
Al	0.009	"
Fe (by diff.)	99.97	"

In case a very smooth deposit is desired, this can be obtained by adding 6 g. ammonium oxalate per liter of electrolyte. However, this addition will cause the deposit to contain more carbon than without it (about 0.02 to 0.03 percent). Furthermore, the oxalate decomposes gradually and has to be replenished from time to time.

Nickel.

Electrolytic nickel can now be obtained on the market, made by the International Nickel Co. A sample analysis follows:

C	0.03	percent
Mn	Trace	"
P	0.001	"
S	0.006	"
Si	0.040	"
Fe	0.250	"
Ni (diff.)	99.673	"

Cobalt.

Cobalt can be prepared by reduction of the oxide, according to the method worked out by Dr. H. T. Kalmus at Queens University,

Kingston, Ontario.[8] Dr. Kalmus gives the following composition of his product:

Co	99.60 percent
Fe	0.33 "
Si	0.097 "
C	0.13 "
P	0.0047 "
S	0.020 "
Al	0.016 "
Ni	None "
Ca	None "

Fairly pure cobalt has been put on the market by the Goldschmidt Thermit Co., a sample analyzing as follows:

C	Trace
Mn	Trace
P	0.008 percent
S	0.058 "
Si	0.250 "
Ni	0.970 "
Cu	0.15 "
Fe	0.60 "
Al	0.07 "
As	0.03 "
Ag	None "
Co (by diff.)	97.864 "

Silicon.

This can be obtained from the Carborundum Company in the form of alloy containing iron in various amounts. The purest grade showed analyses as follows:

Si	94.8 percent
C	0.2 "
Mn	None "
Al	2.0 "
Fe	3.0 "

For the purpose for which silicon is generally used, namely, to raise the resistance of the iron, the above impurities are of little consequence, but for the best results the carbon content is too high. In such cases it is necessary to resort to methods of reduction without the use of carbon.

[8] Canadian Dept. of Mines, Bulletin No. 250 (1913).

Aluminum.

Commercial aluminum from The Aluminum Co. of America is sufficiently pure for most purposes considered here. A typical analysis is as follows:

Si	0.210	percent
Cu	0.100	"
Fe	0.170	"
Mn	< 0.001	"
Ni	None	"
Zn	None	"
Al (by diff.)	99.52	"

Titanium.

Metallic titanium is being made, and will probably be on the market in the near future. Until then, ferro-titanium will have to be used. A sample of the Goldschmidt Thermit Co. product has the following analysis:

Ti	24.00	percent
Si	1.86	"
Fe	68.80	"
Mn	0.83	"
C	0.09	"
S	0.02	"
P	0.006	"
Cu	None	"

Manganese.

This can be obtained from the Goldschmidt Thermit Co., containing a small amount of iron and silicon and practically carbon free. A sample of this product analyzed as follows:

Si	1.85	percent
Pb	Trace	"
Sn	0.007	"
Cu	0.063	"
Fe	1.822	"
Al	0.482	"
Cr	0.750	"
Ni	0.194	"
Mn (diff.)	94.83	"

Vanadium.

Can at present be obtained only as ferro-vanadium. A sample of the Goldschmidt Thermit Co. product gave the following analysis:

Si	0.85	percent
P	0.04	"
S	0.13	"
Ni	None	"
Cr	None	"
Al	4.82	"
Mn	0.17	"
Cu	0.01	"
Fe	53.40	"
V	40.20	"

Boron.

Boron can be reduced to pure metallic form, but only with great difficulty. In the form of suboxide (B_7O), or suboxide flux (containing 8 percent B_7O), it has been extensively used as a deoxidizer for copper.[9] The suboxide furnished the writer while at the University of Illinois by Dr. Weintraub analyzed 82.68 percent boron and may therefore be considered as pure B_7O. Ferro-boron may be obtained from the Goldschmidt Thermit Co.

Carbon.

Carbon can be obtained nearly 100 percent pure from sugar, or if less refined material is required, from charcoal or graphite.

Magnesium.

Can be had at present from a number of chemical companies, the best product containing 99 percent Mg., or even better.

Tantalum.

This is sold by the Goldschmidt Thermit Co. as ferro-tantalum, with a tantalum content of 50 to 60 percent.

Zirconium.

The metallic form can not as yet be obtained in the open market, but can be made in the laboratory by reducing the pure oxide, which is sold by the Foote Mineral Company.

[9] E. Weintraub, Trans. Am. Electrochem. Soc., 16, 165 (1909); 18, 207 (1910).

Uranium.

Can be obtained from the Standard Alloy Company in the form of 30 to 40 percent ferro-uranium.

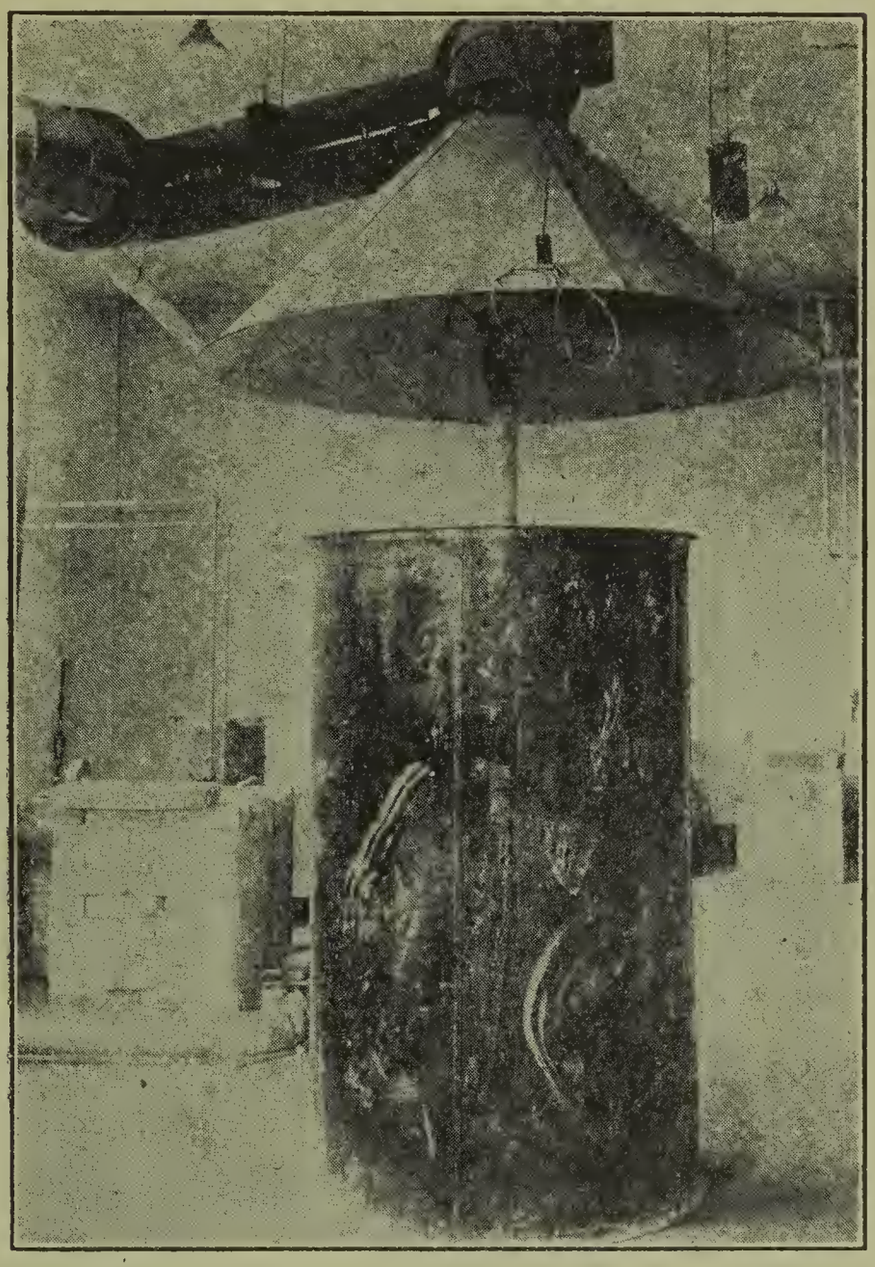


FIG. 2. Arc Furnace for Fusing MgO. Westinghouse Research Laboratory.

III. PREPARATION OF ALLOYS.

The second step in the process of preparing pure alloys is the melting of the pure materials in such a way as not to contaminate them. This means that the crucibles must contain no elements that will be dissolved by the iron, and that the atmosphere in the furnace must be such as not to contribute impurities.

Crucibles.

The crucibles used at the Westinghouse Research Laboratory are made from magnesia. The raw Grecian magnesite calcined at 1,000° C. is first fused in the arc furnace shown in Fig. 2. The tank is filled with magnesite and the arc started with an input of 70 kw. As the magnesia fuses the upper electrode is gradually raised until, at the end of the operation requiring 10 to 12 hours, there is a mass of fused material in the center of the furnace of about 250 kg. The energy consumption per kg. is thus nearly 3 kw. hours. After cooling, the fused magnesia is separated from

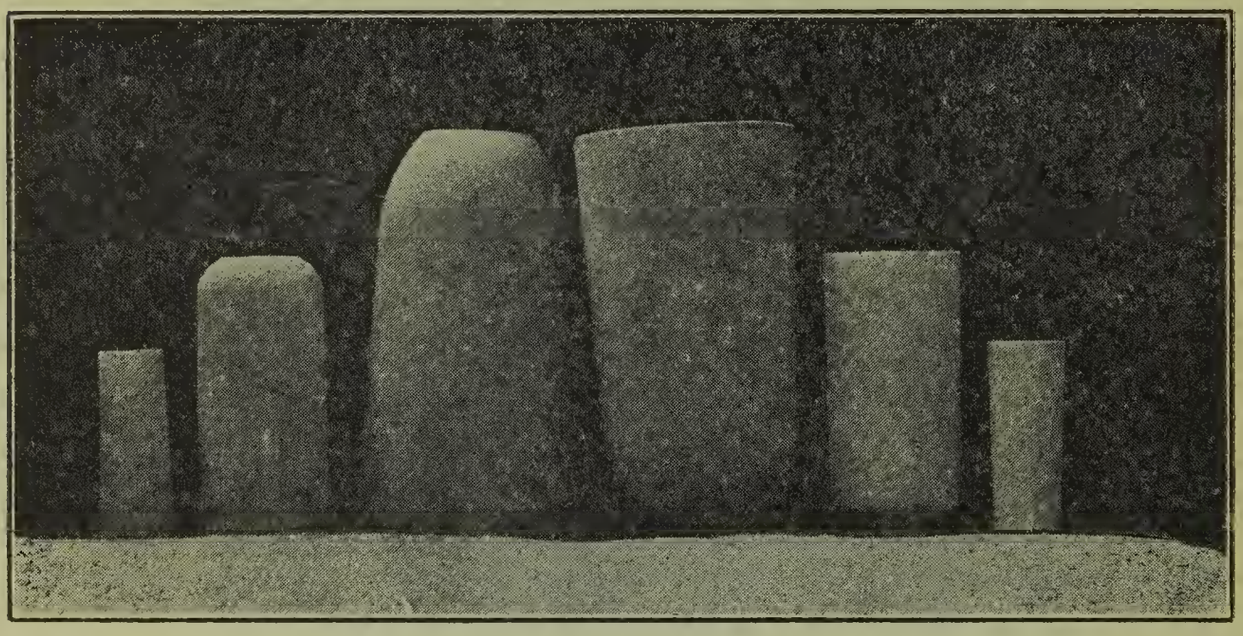


Fig. 3. Magnesia Crucibles Made at Westinghouse Research Laboratory. Tallest crucibles are 7 in. (18 cm.) high, smallest 3 in. (8 cm.) high.

the sintered magnesia and ground to the desired fineness. It has been found by experiment that the best crucibles are obtained using all the magnesia that goes through a 20-mesh screen, although the finer material makes a nicer-looking crucible. The ground magnesia is mixed with a binder consisting of hydrated magnesia, $Mg(OH)_2$, made in a ball mill by mixing together 600 g. calcined magnesite and 1,000 g. water. Magnesium chloride can be used instead of the hydrate and some prefer sodium silicate, but it is believed that the crucibles made with the latter are less refractory than if made with the hydrate or chloride, on account of the silica introduced. The crucibles are pressed in steel molds

under a pressure of about 25 tons. Fig. 3 is a photograph of the various crucibles made. After drying for a day the crucibles are baked in a carbon plate resistance furnace at a temperature of about 1,800° C. Six of each size crucible can be baked at the same

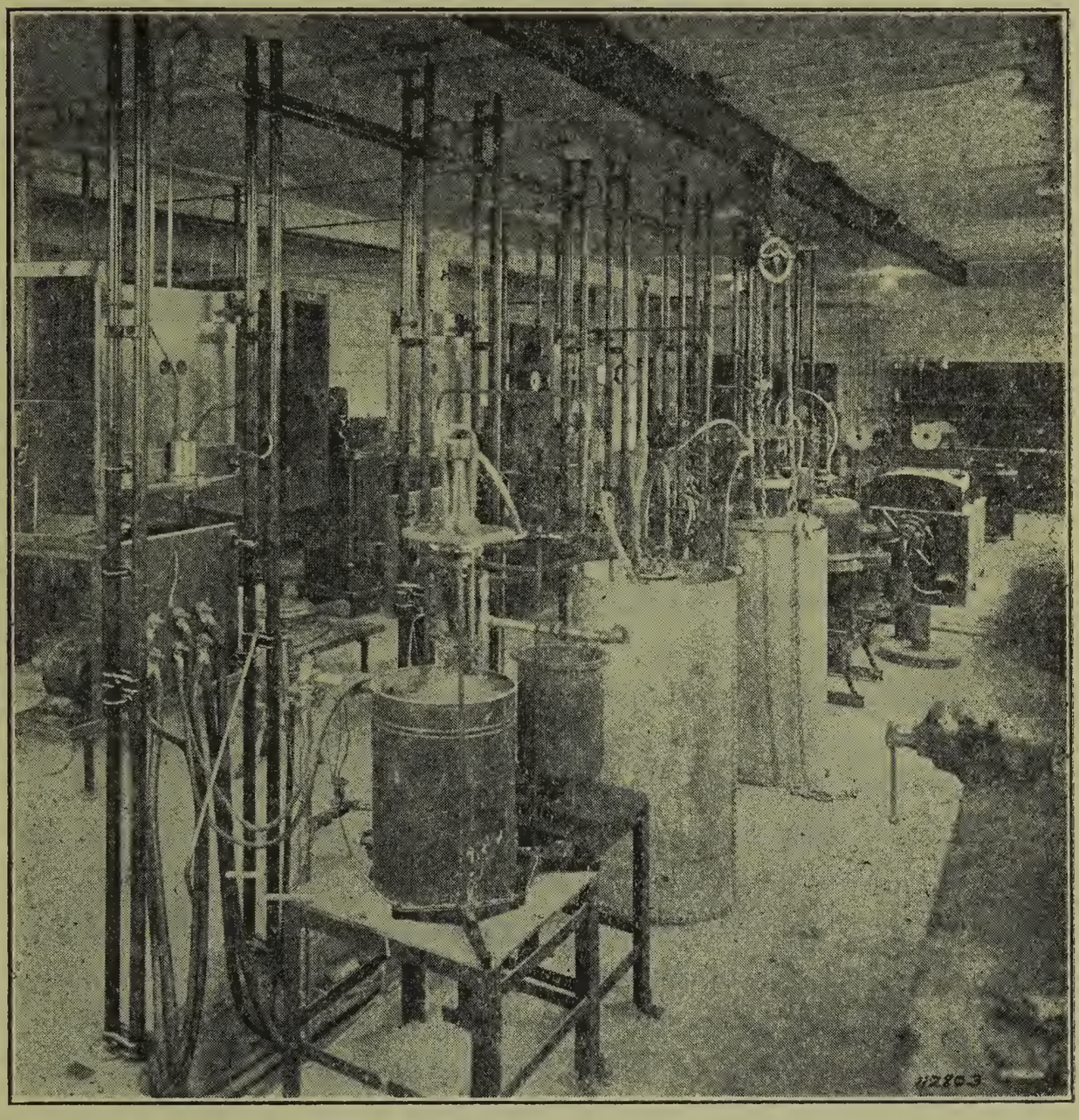


FIG. 4. General View of Furnace Room. Westinghouse Research Laboratory. Four Vacuum Furnaces to the left. Carbon Plate Resistance Furnace to the right.

time, placing them in nests upside down on a magnesia cover. The energy consumption is about 30 kw. for 5 hours followed by 60 kw. for another five hours, making a total of 450 kw.-hours for 18 crucibles with covers. After baking, the crucibles analyze 0.01 percent carbon. The furnace is shown at the right in the

general view of the furnace room in Fig. 4. The lining is made up of three layers of bricks, Sil-o-cel along the outside, firebrick in between, and magnesia brick along the heating chamber. The chamber is covered with graphite plates and these in turn by powdered magnesia. The cover is made from firebrick and Sil-o-cel, and makes a good joint with the main part of the furnace. The water-cooled electrodes are protected from excessive oxidation by being enclosed in drums at each end of the furnace, and these can be so adjusted as to put more or less pressure on the carbon plates that form the heating elements and thus regulate the input.

Furnaces.

Various types of furnaces are used for melting the alloys. Three of these furnaces have to be operated in vacuo or in an inert gas, one furnace can be operated either under ordinary atmospheric conditions or in vacuo, and one can be operated only under ordinary atmospheric conditions. Four of the furnaces use graphite for the heating elements, while one can use either tungsten or molybdenum. In all of the furnaces the behavior of the charge can be observed through windows in the top. The main joints of the vacuum furnaces are made air tight by means of solder or wax that is melted in place by means of resistance ribbon wound around them, and all bushings are made air tight by means of high melting wax.

The vacuum system consists of two units with separate lines of copper tubing having outlets to two valves at each furnace in such a way that any two furnaces can be operated at a time. "Trimount" vacuum pumps are used, exhausting into the main vacuum system of the building. The latter maintains a pressure of 2 to 5 mm., while the Trimount pumps will give as low as 0.0005 mm. under the most favorable conditions. With a furnace connected and iron molten, a pressure of 0.5 mm. can be maintained under favorable conditions.

The electrical equipment also consists of two separate units, so as to serve any two furnaces at a time. Each unit consists of a transformer, induction regulator, and switchboard, so arranged

that any voltage can be obtained from 7½ to 90 volts, and 180 volts by placing the two boards in series. The capacity is 2,000 amperes per unit at the low voltages, 1,000 amperes at intermediate voltages, and 750 amperes at the higher voltages.

In the vacuum furnaces, even with graphite heating elements, it is possible to melt iron without contamination provided the heating element is not heated sufficiently to evaporate the carbon. Under atmospheric pressure, however, iron will absorb as much as 0.10 percent carbon from the CO gases present.

A small amount of refining can be carried out in the vacuum furnaces if necessary. Thus, carbon can be practically eliminated by the addition of iron oxide to the charge. The reaction in its simplest form

$$C + FeO \leftrightarrows CO + Fe$$

takes place from left to right as long as the CO is removed. The equilibrium appears to be reached with a carbon content of about 0.01 percent. Aluminum, titanium and vanadium can also be eliminated by means of iron oxide. On the other hand, silicon, boron and manganese can be only partially removed, because of their high affinity for iron. *Vice versa,* iron can be completely deoxidized by means of any of the above elements. Carbon, aluminum, titanium or vanadium will accomplish the deoxidation without leaving any but traces of themselves in the iron, unless they are added in excess, carbon going off as CO and the others passing as oxides to the top of the bath and into the slag. Silicon, boron and manganese, however, will partly combine with the iron at the same time as they combine with the oxygen. It is further claimed for titanium that it will remove any nitrogen dissolved in the iron.

The alloys thus made are rolled or forged into the desired shape for testing or for other purposes. While it has been found that this operation can be performed under ordinary forging conditions without danger of contamination, it is well known that the product must be thoroughly annealed afterwards to eliminate the strain caused by the mechanical working. Furthermore, for commercial

materials containing some impurities, annealing at high temperatures has been shown to increase the size of the crystals, with a highly beneficial effect in lowering the hysteresis loss. This is particularly true for silicon steel. It is doubtful, however, whether annealing has any such effect upon pure iron and iron-silicon alloys free from other impurities, as it has been shown that the size of the crystals in the latter case is practically unaffected by annealing. Whether the beneficial effect of annealing is partly

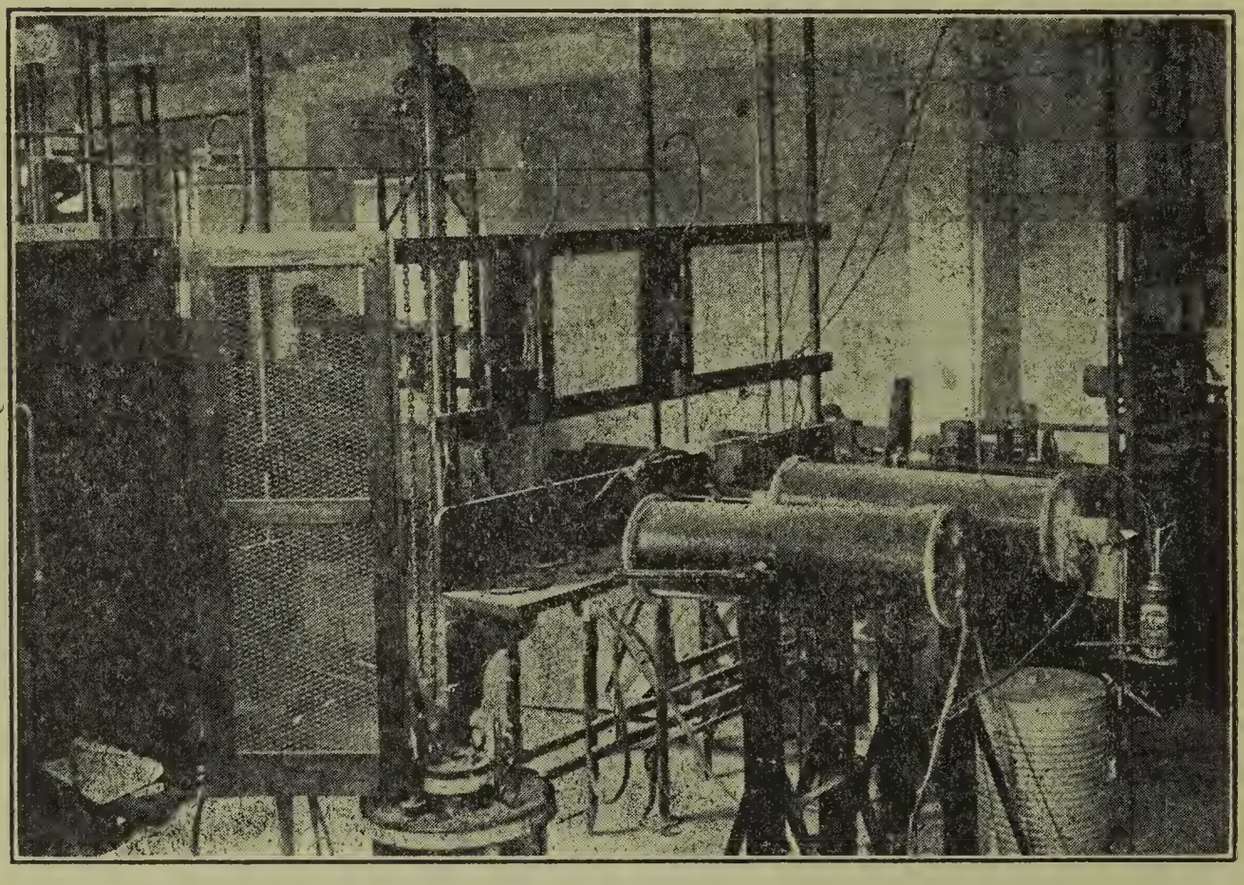


FIG. 5. Annealing Furnaces. Westinghouse Research Laboratory. Hoskins Furnace Outside Picture to the right.

due to the converting of all the *beta* and *gamma* iron into *alpha* iron is also a debated question. That it is not chiefly due to this cause in the case of uncontaminated products is shown by the fact that most of the improvement in permeability and hysteresis loss for such materials takes place below the α to β transformation point.[10]

Fig. 5 shows the battery of annealing furnaces at the Westinghouse Research Laboratory. Two of them are tube furnaces for

[10] Yensen, Bulletin No. 72, Eng. Exp. Station, Univ. of Illinois.

annealing rods, bars or strips. The tubes are of fused quartz and can be closed at both ends with rubber stoppers for operation with vacuum or various gases on the inside. The nichrome heating elements are wound on alundum tubes and can be heated to give a temperature of 1,100° C. inside the quartz tubes. The furnaces can be swung into a vertical position for filling or for dropping

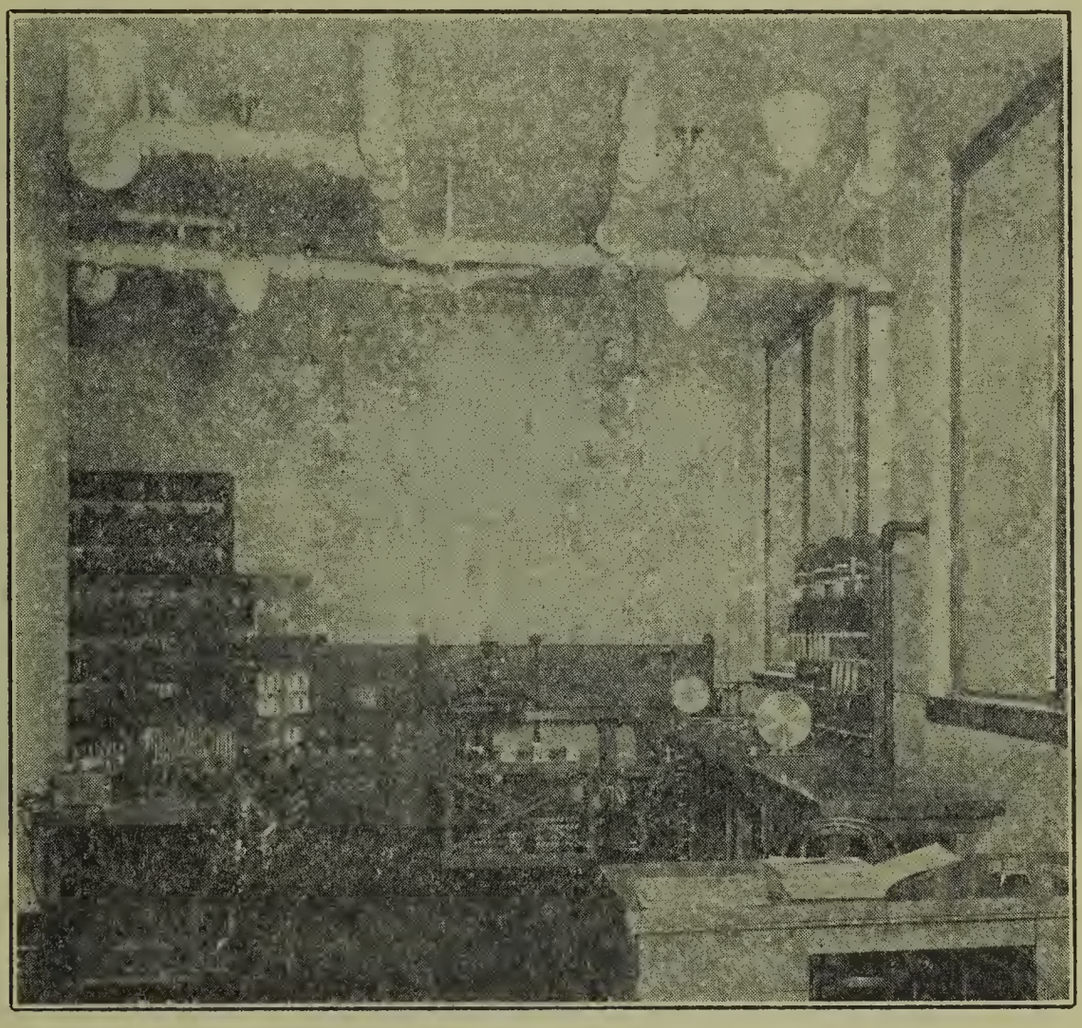


Fig. 6. Magnetic Testing Laboratory. Westinghouse Research Laboratory.

test pieces into a quenching bath. For annealing, the test pieces are placed in powdered magnesia and attached to a quartz pyrometer tube projecting out through one of the rubber stoppers. A pressure of 0.05 mm. can be maintained in the tubes during the annealing. Another furnace, similarly wound, is operated in a vacuum tank in a vertical position and is used for annealing circular punchings or solid rings too large to go into the tube furnaces. Finally, a Hoskins furnace is used for all other purposes not

requiring vacuum treatment. The electrical equipment for these furnaces consists of three induction regulators, so that three furnaces can be operated simultaneously.

The magnetic testing laboratory, shown in Figs. 6 and 7 is equipped with the latest and most approved apparatus for magnetic testing, consisting of a modified Burrows outfit for permeability

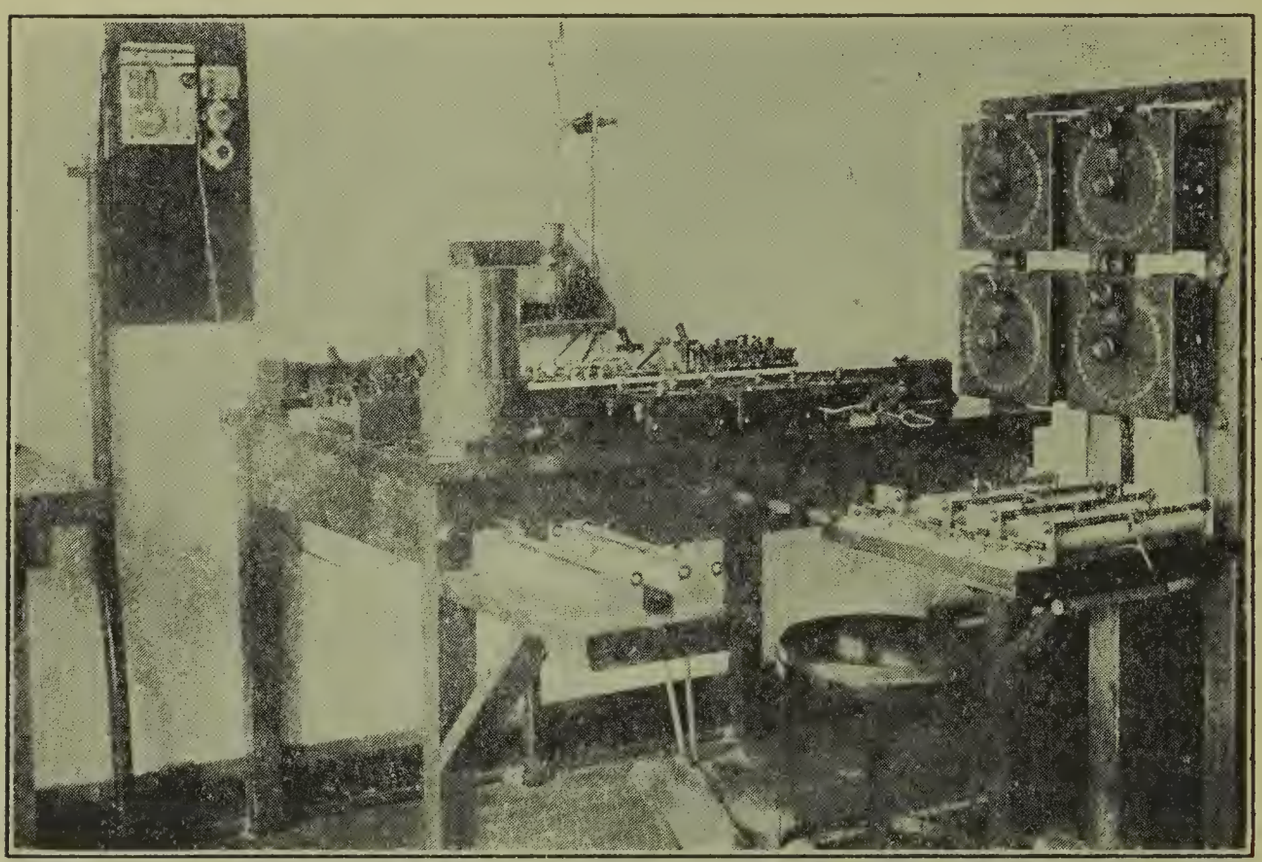


FIG. 7. Magnetic Permeability Testing Outfit. Westinghouse Research Laboratory.

and hysteresis tests of bars, rods, and strips, which can also be used for testing of ring samples; Epstein apparatus for testing iron losses of sheet material with auxiliaries for obtaining various wave shapes; an outfit for testing magnetic uniformity of rods; and, in an adjoining room, apparatus for testing electrical resistance and temperature coefficients.

Research Laboratory,
Westinghouse Electric & Manufacturing Co.,
East Pittsburgh, Pa.

Aug. 13, 1917.

DISCUSSION.

W. RUDER[1]: Mr. Yensen's work, which he has published at different times, as to the exceptional permeability values and the low-watt losses he has obtained in his vacuum-fused irons, is certainly a commendable one.

The value of the *extreme* purity in magnetic materials is evidenced by the very large difference obtained in the magnetic testings of the extremely pure material, such as has been obtained by vacuum fusion of the electrolytic iron, and the magnetic tests obtained from practically pure irons such as good Norway iron or American ingot iron. Although the vacuum fusion of electrolytic iron on a large scale appears to be a difficult thing, yet Mr. Yensen has set, by his work, new limits to our aspirations, and we can now hope to achieve greater results in the preparation of our commercial products.

PRESIDENT C. G. FINK[2]: I should just like to add that I do not agree with Mr. Yensen on his melting point determination of cobalt. I do not believe you can get absolutely pure cobalt in a vacuum furnace made up with a carbon resistor. Cobalt takes up carbon very readily, and the melting point is lowered over 100 degrees by the merest trace of carbon. The melting point of cobalt, as determined in our laboratory two years ago, is 1,610° C., which is about 110° higher than Mr. Yensen's value.

T. D. YENSEN: If I understand the chairman correctly, does he state that cobalt, and iron alloys, cannot be melted in a carbon-resistance vacuum furnace without absorbing carbon?

PRESIDENT C. G. FINK: That is correct.

T. D. YENSEN: I do not remember that we had our iron-cobalt alloys analyzed for carbon. The chief importance of cobalt is not in attaining exceedingly high permeabilities at low flux densities, but rather in attaining high saturation, or high permeabilities at exceedingly high densities. The iron-cobalt alloy, Fe_2Co, was first obtained by Dr. Weiss, of Zurich.

The saturation value of this alloy is 10 to 13 percent higher

[1] General Electric Co., Schenectady, N. Y.
[2] Director of Research, Chile Exploration Co., New York City.

than that of pure iron. I do not believe that a small trace of carbon in this case would be very serious, but it may be possible to get higher values.

PRESIDENT C. G. FINK: Have you ever tried to melt the ferro-cobalt in an atmosphere other than CO?

T. D. YENSEN: No, I have not.

PRESIDENT C. G. FINK: Don't you think you would get higher saturation values if you eliminated the last traces of carbon?

T. D. YENSEN: It is possible that we may. Attempts will be made to melt the iron-cobalt alloys in other kinds of furnaces. There are other types available besides the carbon resistor type.

T. D. YENSEN (*Communicated*): Since the discussion of my paper in October, analyses have been obtained on the carbon content of a few iron-cobalt alloys containing from 1 to 15 percent cobalt, prepared in a carbon resistor vacuum furnace. According to these analyses the carbon content is

0.016 percent for the 1 percent alloy
0.009 percent for the 2 percent alloy
0.005 percent for the 3 percent alloy
0.035 percent for the 4 percent alloy
0.009 percent for the 5 percent alloy
0.013 percent for the 10 percent alloy
0.005 percent for the 15 percent alloy

Judging from this result it should be feasible to melt iron-cobalt alloys in a carbon resistor vacuum furnace without contamination by carbon. I do not think that any marked improvement can be obtained in the saturation values of these alloys by a further elimination of carbon.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

A METHOD FOR THE COMMERCIAL ANALYSIS OF FERRO-SILICON

By Russell E. Lowe.*

[Abstract.]

The method of procedure described involves the decomposition of the alloy by means of nitric and hydrofluoric acids, and the determination of those elements that are generally called for in specifications for the 50 percent alloy; namely, manganese, sulphur, phosphorus, iron, aluminum, and silicon.

In the sulphur and phosphorus determination a method is presented for the necessary removal of the last traces of fluorine without the use of sulphuric acid.

The method, as given, has all of the accuracy of the older fusion methods while at the same time it is more rapid, easier to handle, and saves platinum.

Some remarks are appended upon the probable volatilization of phosphorus from samples of ferro-silicon kept nearly ten years in screw-topped glass jars.

This analytical procedure is the result of an extended series of experiments, carried on with the purpose of evolving a rapid and accurate method for the analysis of ferro-silicon without the aid of the powerfully oxidizing fluxes at present used. By "commercial analysis" is meant the quantitative determination of sulphur, phosphorus, manganese, iron, aluminum and silicon, since these elements are the ones generally specified in contracts for the 50 percent alloy.

The older procedures for the analysis of ferro-silicon require a flux consisting of 20 grams of sodium carbonate and 4 grams of potassium nitrate to break down a 1-gram sample of the alloy.

* Chemist, The FitzGerald Laboratories, Inc., Niagara Falls, N. Y.

A fusion of this character is decidedly destructive to platinum, and in addition requires almost constant supervision because of its tendency to creep over the side of the crucible. Another, and a great fault of the fusion method is that it is slow. Because of the minute quantities of impurities present in the alloy, a gram sample at least must be used. This means the tedious handling of a large volume of silica, and involves a second fusion since it is impossible to wash silica absolutely free from the heavy metals.

The following method of procedure has been tried on several samples of ferro-silicon that were previously analyzed by an outside laboratory of recognized standing, and in practically all cases the agreement was found to be within the limits of experimental error. The method is recommended as being simple, accurate, rapid, and saving of platinum.

DETERMINATION OF SULPHUR AND PHOSPHORUS.

Mix sample thoroughly and powder in a diamond mortar until it passes through a 100-mesh screen. Weigh about 1 g. of the powdered sample into a 100 c.c. platinum dish, add 25 c.c. of concentrated nitric acid, cover with platinum foil and place on a boiling-water bath. When the contents of the dish have reached the temperature of the bath add a few drops of hydrofluoric acid. As soon as the resulting violent reaction has ceased add a second portion of the acid. Continue this procedure until only a slight residue is visible on the bottom of the dish, then add about 5 c.c. more of hydrofluoric acid. Remove the watch glass, carefully washing any matter adhering to it into the platinum dish with a stream of hot water, and evaporate the solution to dryness. Add 10 c.c. of concentrated nitric acid to the contents of the dish and repeat evaporation on water bath. Dissolve residue in dish with 50 c.c. of a 5 percent solution of nitric acid, heating to hasten the process.

Add to the dish a concentrated solution of sodium carbonate in small portions and with constant stirring until it is present in slight excess, then transfer the contents to a glass beaker and dilute with hot water to about 200 c.c. Heat contents of beaker to boiling and allow to settle. Filter solution through a 12.5 cm. filter, washing the precipitate five times with hot water.

Sulphur:—The filtrate contains the sulphur, and to it is added an aqueous solution of calcium chloride until the precipitation of calcium fluoride and calcium carbonate is complete. Boil the solution for a few minutes, allow the precipitate to settle, filter through a 12.5 cm. filter paper. Wash the precipitate five times with hot water.

Acidify filtrate from the calcium chloride precipitation with hydrochloric acid. Heat the solution to boiling and add 25 c.c. of boiling 6 N barium chloride. Allow solution to stand in a warm place for one hour, then filter through a 9-cm. No. 42 Whatman filter, or its equivalent. Wash filter with hot water until it is free from chlorides.

Ignite this filter in a weighed platinum crucible, cool in dessicator and weigh the sulphur as barium sulphate. Calculate to sulphur. As in all sulphur determinations it is advisable to run a blank carrying out the procedure as given, omitting of course the sample, otherwise results are apt to be too high.

Phosphorus:—The sodium carbonate precipitate contains the phosphorus, as iron *phosphate.* This precipitate is dissolved from the filter with hot nitric acid, sp. g. 1.13 (approximately 1 volume of concentrated nitric acid to 3 of water). 50 c.c. of the acid will be found ample if it is poured through the filter several times. When the precipitate is completely dissolved the filter is washed, first with a 1 percent solution of nitric acid, then with hot water until it is free from iron.

The solution is now heated to boiling, with the addition of a few drops of a concentrated solution of potassium permanganate, and kept at this temperature for about five minutes. Add a solution of ferrous sulphate in water, acidified with sulphuric acid, until any precipitate of manganese dioxide dissolves and the solution becomes quite clear.

Remove solution from the source of heat and add 50 c.c. of ammonium molybdate solution. Allow to stand for 30 minutes, filter through a 9-cm. filter paper, then wash first with 1 percent nitric acid solution, second with hot water until the yellow precipitate is free from iron.

After the method of J. O. Handy,[1] transfer the filter paper and yellow precipitate to a glass beaker, add a definite volume of

[1] Blair, Chemical Analysis of Iron, 7th ed., p. 104.

sodium hydroxide solution that has been standardized against a steel of known phosphorus content, and titrate for the excess of alkali with a standardized solution of nitric acid. Calculate to phosphorus.

DETERMINATION OF MANGANESE, IRON, AND ALUMINUM.

Weigh about one gram of the finely powdered sample into a platinum dish and proceed with the decomposition of the alloy in a manner identical with that given in the sulphur-phosphorus procedure, up to and including the first evaporation to dryness.

Cover residue in the dish with 25 c.c. of a 1:5 solution of sulphuric acid. Evaporate solution almost to dryness. Cool. Add 25 c.c. of distilled water, raise contents of the dish to the boiling point, then transfer to a glass beaker. Add 100 c.c. of water, a few drops of concentrated hydrochloric acid and boil until solution is complete. Determine manganese, iron and aluminum in this solution in the regular manner, as given in Blair's Analysis of Iron and Steel, or any standard work on qualitative analysis.

DETERMINATION OF SILICON.

Silicon is calculated by summing up the percentages of the elements determined and subtracting from 100.

For an extremely rapid but approximate determination of silicon, accurate within 0.5 percent, weigh 0.25 g. of the sample into a weighed platinum dish. Decompose with mixed acids as before. Evaporate solution to dryness. Ignite dish at red heat, cool in dessicator and weigh. Assume residue to be entirely composed of iron oxide. Calculate to iron and subtract this value from the weight of the sample, calling the difference silicon.

Notes on the Analysis.

1. Nitric acid must be added first to prevent the volatilization of iron, sulphur, phosphorus, etc., as fluorides.

2. By keeping the solution hot it is possible to maintain a much more even rate of decomposition. When hydrofluoric acid is added to a cold solution the reaction is very apt to start with such violence as to eject the contents of the dish.

3. The treatment with sodium carbonate prior to transferring the solution from platinum to glass is necessary because of the

traces of free hydrofluoric acid invariably present in the acid solution. For the determination of sulphur and phosphorus it is obviously impossible to make use of a sulphuric acid evaporation to remove the last traces of fluorine. Likewise, the contents of the dish cannot be evaporated to dryness and baked owing not only to the danger of volatilizing phosphorus, but also to the difficulty of getting the ignited oxides back into solution.

By the addition of sodium carbonate, the traces of fluorine remaining are converted to the fluoride and this is later removed by precipitation with calcium chloride in the sulphur determination. Before this method was adopted the etching of beakers and cover glasses was quite noticeable, while the presence of gelatinous silica made filtrations most difficult.

4. It is to be noted that in no case is there an evaporation of a ferric chloride solution in contact with platinum. This eliminates another source of loss of that expensive metal.

THE VOLATILIZATION OF PHOSPHORUS FROM FERRO-SILICON.

In the course of the experiments for evolving the rapid method for the commercial analysis of ferro-silicon just described, the analytical procedures as worked out were tested on several samples of ferro-silicon that had been previously analyzed by the Pittsburgh Testing Laboratories by the regular method of procedure.

The new method gave excellent checks in all but the phosphorus determinations, and here, invariably, percentages were obtained that were approximately half of that quoted, as the following data shows:

	Percent of Phosphorus	
No. of Sample	P. T. L.	Rapid Method
410	0.022	0.0103
411	0.044	0.022

Feeling quite certain that the fault did not lie in the method but in the sample itself, the author repeated his work, this time using an oxidizing flux of carbonate and nitrate to break down the alloy. The results thus obtained also proved to be about half the reported values for phosphorus.

Sample No. 411 was then returned to the Pittsburgh Testing Laboratories for a new determination of phosphorus. While

waiting for their report, an investigation of the laboratory files showed that the first analysis had been made in February, 1908, almost ten years previously.

In due time the Pittsburgh Testing Laboratories reported on the phosphorus content of the sample, their value being 0.020 percent, a very satisfactory check upon the value, 0.022 percent obtained by the rapid method.

From the above it seems reasonable to conclude that the volatilization of phosphorus from ferro-silicon is considerable, even when the alloy is kept in a dry place enclosed in screw-topped glass jars.

The FitzGerald Laboratories, Inc.,
Niagara Falls, N. Y.
August 30, 1917.

DISCUSSION.

T. D. YENSEN[1]: I regret very much that Mr. Lowe has left out carbon from his list of elements. We are very much interested in the determination of carbon in silicon, and I should like to ask Mr. Lowe if the direct combustion method will burn all of the carbon out of the silicon so that accurate analyses can be obtained that way?

I should also like to ask Mr. Lowe if there is any means of getting the carbon eliminated from the silicon without at the same time oxidizing the silicon? If this is not possible, is there any way of obtaining silicon that is carbon-free?

R. E. LOWE: In the various samples of ferro-silicon that have come to our attention we have found that the maximum carbon content in the 50 percent alloy is 0.2 percent. So, in working up this method of commercial procedure, we neglected carbon, as it was rarely called for in specimens that came to us for analysis. We found that it was impossible to determine carbon by the direct ignition of the alloy in oxygen, it being necessary to mix the sample with a considerable quantity of red-lead before making the determination by the regular oxygen combustion method. This

[1] Research Laboratory, Westinghouse Elec. & Mfg. Co., East Pittsburgh, Pa.

red-lead method is decidedly disagreeable, and I do not believe that it is particularly accurate, as one cannot use a large sample. The combustion is so furious that if a gram sample is taken for analysis the combustion tube is very apt to be ruined. We did find that by taking the very slight residue remaining after the nitric-hydrofluoric acid treatment, and weighing it as mixed carborundum and graphite, then igniting and weighing again, it is possible to determine the carbon present as silicon carbide and as graphite.

I do not know of any method of producing carbon-free silicon in the electric furnace, or of a method of treating silicon to render it carbon-free.

R. E. Lowe (*Communicated*): It has been found advisable to discontinue the practice of covering the platinum dish with a watch-glass during the nitric-hydrofluoric acid treatment, owing to the fact that a determination of calcium may at times be called for, and chemical glassware is usually made from a lime glass.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

INFLUENCE OF TEMPERATURE OF MEASUREMENT ON THE ELECTRICAL RESISTIVITY AND THERMO-ELECTROMOTIVE POTENTIAL OF SOLUTES IN STEELS.

By Edward D. Campbell.*

It is now more than a century since Proust, in France, first stated that white cast iron should be regarded as a solidified solution of carbides of iron in iron, and more than a half century since Matthiessen, in England, also maintained that steel and other alloys should be considered as solid solutions, the electrical properties of which are dependent upon the chemical composition. The solution theory of metals is now nominally accepted by most metallurgists and physicists and yet the question may be raised as to how many experimenters have accepted the essential unity of mechanism of metallic and aqueous solutions, and have based their experimental work on this conception.

In studying the properties of ordinary solutions the part played by the solvent is clearly differentiated from that played by the solutes. It is those solutes which are in solution that are capable of reacting with electrical energy transporting it as an electric current or with radiant energy such as light, producing absorption or refraction. In 1915 (Journal of Iron and Steel Institute, 1915, No. II, p. 164) the author brought forward experimental evidence to show that the electrical resistivity of steels is a function of the concentration of the carbides or other solutes in solution. A year later (Journal of Iron and Steel Institute, 1916, No. II, p. 268, and Transactions of The Faraday Society, Vol. XII, 1917, p. 274) the dependence of the electrical resistivity on the concentration of the solutes was confirmed; and it was further shown that if carbides are the only solutes present the thermo-electromotive potential of these carbides is also a function of the concentration. The hypothesis was advanced at that time that it is the molecules of solutes in solution which are capable of

* Professor of Chemistry and Director of the Chemical Laboratory, Univ. of Mich.

reacting with electrical energy transforming it into heat, thus causing electrical resistivity. Electrical conductance seems to take place almost wholly through the solvent, while the total resistivity seems to be made up of two components—first, that portion due to the solvent, and, second, that due to the solutes in solution. It has been known for many years that the temperature coefficient of electrical resistivity of nearly all strictly pure metals is approximately constant between 0° C. and 100° C., while the temperature coefficient of impure metals and alloys is always lower than that of pure metals, often being very small or even negative. In determining the temperature coefficient of resistivity it is customary to determine the specific resistance of the metal as a whole at two or more temperatures and calculate the change in specific resistance to a percentage basis of the specific resistance at a standard temperature. No attention is paid to the solution theory of metals, for the real acceptance of this conception would require that the total specific resistance be regarded as made up of two components—first, that due to the solvent, and, second, that due to the solutes, with the presumption that the laws governing the two portions might differ.

If, under the solution theory, the total specific resistance is to be regarded as made up of two components, it would be only reasonable to expect that under the same conception the thermo-electromotive potential of a metallic solution might also be regarded as made up of two components—that due to the solvent and that due to the solutes in solution.

The principal objects of the present investigation, the laboratory work of which was carried on by William C. Dowd, M.S., were to bring forward some further experimental evidence of the essential unity of mechanism of metallic and aqueous solutions and to show that while the electrical properties of metals are dependent upon the chemical constitution, the effect of changes in temperature of measurement on that portion of the properties due to solvent are not the same as those controlling that portion due to solutes in solution.

EXPERIMENTAL.

Steel is one of the best materials with which to study the solution theory of metals because the solvent iron is capable of exist-

ing in at least two allotropic forms, and certain solutes, noticeably carbides, are quite readily soluble in iron in the *gamma* condition, although only slightly soluble in *alpha* iron. It is the difference in the solvent and dissociating power of iron in its various allotropic forms that enables the concentration of certain solutes, particularly carbides, to be controlled by heat treatment; thus modifying the chemical constitution without change in the ultimate composition. The stock samples of steels used in the present research were furnished by the courtesy of the American Rolling Mill, the Bethlehem, the Carpenter, the Halcomb and the Midvale Steel Companies, and have the composition shown in Table I, in which the steels are arranged in order of the carbon content.

TABLE I.

Steel	Carbon Percent	Manganese Percent	Phosphorus Percent	Sulphur Percent	Silicon Percent	Copper Percent	Nickel Percent	Chromium Percent	Tungsten Percent
INI	0.018	0.016	0.005	0.018	....	0.018	....	.....	.....
SN3	0.104	0.122	0.03	0.025	3.649	....	....	.....	.....
M8	0.326	0.405	0.013	0.026	0.162	....	4.48	.....	.....
H12	0.61	0.25–0.35	0.020	0.020	0.20–0.30	....	....	0.20–0.30	5.00–5.25
C5	0.945	0.189	0.013	0.016	0.155	....	....	.....	.....
SN1	1.11	0.40	0.020	0.038	1.20	0.06	0.31	0.348	.....
MN1	1.15	11.57	0.055	0.023	0.25	....	....	.....	.....
INC	1.18	0.016	0.005	0.018	....	0.018	....	.....	.....

With the exception of the sample marked INC, stock samples of all the steels were annealed by heating over night in an electrically heated furnace to the temperature given in each case and allowing the steel to cool with the furnace for twenty-four hours. From the stock bars, which were thus very carefully annealed under conditions which would completely prevent oxidization, small experimental bars 6 millimeters square and 15 centimeters in length were machined out. These experimental bars were used for studying the influence of heat treatment on the electrical properties.

The sample marked INC was prepared by carburizing small bars of INI, twelve of these small bars being carefully packed in a carburizing mixture made up of ¼ barium carbonate, ¼ animal charcoal and ½ wood alcohol. The muffle containing the bars packed in the carburizing mixture was heated over night in

an electrically heated furnace, the maximum temperature reached the next day being 969° C. The temperature was then maintained for two days between 960° C. and 945° C., allowed to cool over night to 845° C., maintained one day at 845° C., and the next day held between 820° C. and 800° C., after which the furnace was allowed to cool to room temperature. Analysis of the bars showed 1.18 percent carbon, and a micro-section showed that carburization had proceeded through the entire bar; in fact, the excess cementite tended to be more marked in the center than near the periphery of the bars.

In order to harden the steels, bars were suspended in an electrically heated furnace which had been previously brought to the temperature from which it was desired to quench. The furnace was so arranged that the bars could be suspended in it for an indefinite length of time without any oxidation taking place. The bars were kept in the furnace for one hour to ensure their attaining the same temperature as that of the furnace, which was measured by means of a standard platinum-rhodium thermo-couple, placed within two or three millimeters of the bars. When the bars had come to constant temperature they were withdrawn from the furnace and quickly cooled in a large volume of iced water. The time required to transfer a bar from the furnace to the cooling bath, taken with a stop-watch, averaged four-fifths of a second. The time required to cool from the quenching temperature to a black heat outside was from four-fifths of a second to one second, and until the bars reached the temperature of the cooling-bath from five to six seconds. During the time the bars were being transferred from the furnace to the quenching-bath a film of oxide was formed on the surface, but this was so slight as to constitute only a discoloration.

When the bars had been quenched they were carefully polished and measurements made of the specific resistance at 25° C., and of the thermo-electromotive potential over a temperature gradient of 0° C. to 25° C., the freshly quenched bars being opposed to annealed bars of the same steel. Since it was desired to study the influence of temperature of measurement on the specific resistance and thermo-electromotive potential of the steels and since the solid solution in freshly-quenched steel is distinctly unstable, it was necessary to bring these steels to a state which would be

fairly stable at temperatures up to 100° C. This was done by placing the freshly-quenched bars, after the measurements of specific resistance and thermo-electromotive force at 25° C. had been made, in an electrically heated drying oven maintained at 105° C. and allowing the bars to temper at this temperature from two to three days. After tempering at 105° C. in this way, measurements of the specific resistances were made at 25° C., 40° C., 60° C., 80° C., and 100° C., and also of the thermo-electromotive potentials over the temperature gradients 0° C. to 25° C., 40° C., 60° C., 80° C., and 100° C., the tempered bars being opposed to annealed bars of the same steel.

The resistivities were determined by measuring the fall in potential between knife-edges clamped to the bar 10 centimeters apart, while a current of constant density was flowing through the bar. The fall of potential was compared with that given by a standard resistance which had been calibrated by means of a Siemens and Halske potentiometer and a certified standard cell. The knife-edges are bolted to a small glass plate to ensure constancy of distance apart, and the bars, during the measurements, were immersed in a bath of paraffin oil, the temperature of which could be maintained at any desired point during the measurement. Measurements could be easily duplicated to within less than 0.05 microhm, and it is thought that the values given do not differ more than 0.2 microhm from their absolute value.

The thermo-electromotive force developed over the temperature gradient 0° C. to 25° C., 40° C., 60° C., 80° C., and 100° C., was determined by the following method: Three bars of the same steel and of the same heat treatment were fastened together by means of small pure iron clamps so as to form a triple bar about 43 centimeters in length. One end of this triple bar was clamped to one end of a similar triple bar which in the case of freshly-quenched or tempered bars was made up of three small bars of the same steel in the annealed condition. The thermo-electromotive potential of annealed bars was obtained by opposing to a triple bar of the annealed steels a triple bar made up of pure ingot iron. The compound bars were so shaped that the point of connection of the heat treated and annealed triple bars was immersed in the oil bath also used for specific resistances. By means of small clamps machined out of carefully annealed ingot iron,

pure iron wire leads were attached to the outer ends of the two triple bars. The upper temperature was that of the oil bath in which the connected inner ends of the two triple bars were immersed. The lower temperature of the gradient was obtained by supporting the outer ends of the triple bars in two large assay crucibles filled with crushed ice. A vertical slot 7 or 8 millimeters wide and 5 centimeters deep was sawed in each crucible, the bottom of the slot serving as a support for one end of the triple bar. A small hole was drilled in the bottom of the crucible to prevent accumulation of water and the crushed ice was prevented from falling out through the slot by a small piece of sheet copper, bent so as to hang from the top of the crucible on the inside, but not quite reaching to the bottom.

The ends of the pure leads not connected to the compound bars were attached by means of small connectors to the copper conductors leading to the galvanometer. The connectors joining the iron leads and copper conductors were placed in a small bottle of paraffin oil, the object of this being to ensure both connectors being at the same temperature. The galvanometer used is the same as that employed in measuring specific resistances and the deflections are converted into micro-volts by comparing with those obtained by connecting to a standardized resistance through which a known current was passed. Six micro-volts produced one millimeter deflection on the galvanometer scale, on which by means of a telescope and cross hair, tenths of a millimeter can be estimated. Duplicate determinations could be kept inside of five or six micro-volts, and it is thought that the values obtained are not more than ten micro-volts from their true value.

The specific resistances of the pure iron and seven steels are shown in Table II, in which table the steels are arranged according to their carbon content. In this table the first column indicates the name of the steel; the second, the percent of carbon; the third, the heat treatment; the succeeding five columns give the specific resistances at the different temperatures; while the last column gives the increase in specific resistance from 25° C. to 100° C. The difference in the specific resistance of freshly quenched samples and the same steels after tempering at 105° C., both being measured at 25° C., clearly shows the instability of the solid solution in freshly quenched metal and why it was

TABLE II.

Steel	Percent Carbon	Heat Treatment	Resistivity Measured at 25° C.	40° C.	60° C.	80° C.	100° C.	Increase of Resistivity 25° C.-100° C.
IN1	0.018	Annealed from 778° C.	10.64	11.43	12.49	13.67	14.91	4.27
SN3	0.104	Quenched from 1002° C.	55.39					
		Quenched and tempered at 105° C.	55.22	55.71	56.38	57.10	57.95	2.73
		Annealed from 985° C.	53.37	53.91	54.66	55.46	56.45	3.08
M8	0.326	Quenched from 900° C.	29.53					
		Quenched and tempered at 105° C.	28.60	29.33	30.42	31.64	32.84	4.24
		Annealed	25.29	26.02	27.17	28.41	29.72	4.43
H12	0.61	Quenched from 900° C.	30.39					
		Quenched and tempered at 105° C.	26.48	27.27	28.40	29.72	31.12	4.64
		Annealed from 720° C.	19.15	19.94	21.08	22.35	23.75	4.60
C5	0.945	Quenched from 902° C.	37.48					
		Quenched and tempered at 105° C.	26.15	27.07	28.40	29.79	31.25	5.10
		Annealed from 903° C.	16.96	17.63	18.78	20.07	21.48	4.52
SN1	1.11	Quenched from 1002° C.	68.87					
		Quenched and tempered at 105° C.	63.59	64.45	65.63	66.83	68.05	4.46
		Annealed from 950° C.	41.62	42.36	43.39	44.45	45.72	4.10
MN1	1.15	Quenched from 1002° C.	68.68					
		Quenched and tempered at 105° C.	68.54	69.73	71.55	73.43	75.32	6.78
		Annealed from 810° C.	54.31	55.19	56.70	58.42	60.31	6.00
INC	1.18	Quenched from 902° C.	47.44					
		Quenched and tempered at 105° C.	30.32	31.58	33.31	35.10	37.07	6.75
		Annealed from 905° C.	15.81	16.82	18.23	19.67	21.14	5.33

necessary to temper at 105° C. if consistent results were to be obtained with temperature of measurement as high as 100° C.

Since such a large proportion of the total resistivity is due to solutes in solution and since annealing produces precipitation of

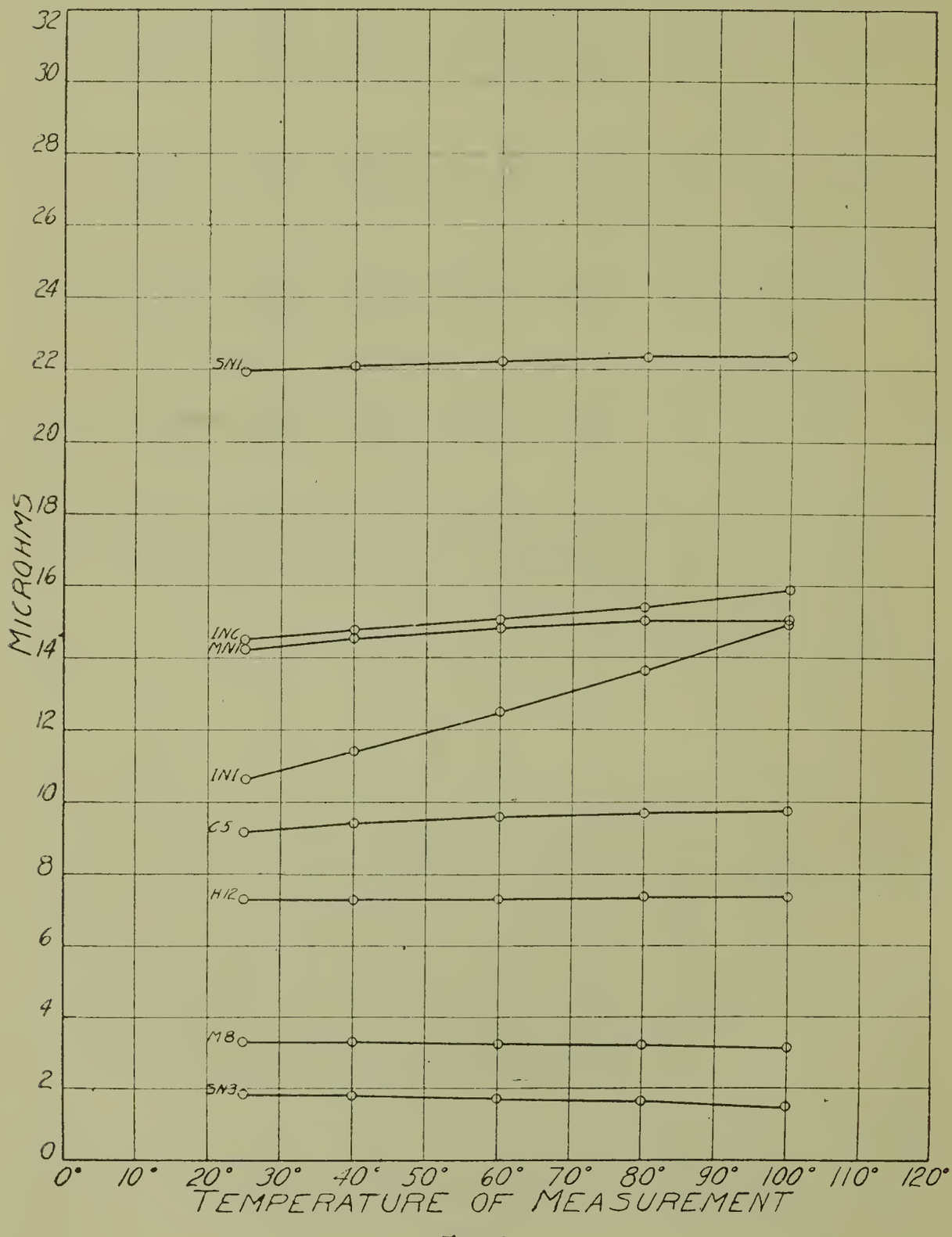


FIG. 1.

some of the solutes, particularly carbides, if we subtract from the resistivity of tempered steel the resistivity of the same metal in

the annealed condition we would have that portion of the electrical resistivity which is due to those solutes which are in solution in the tempered, but not in solution in the annealed, steel. These differences of resistivities are given in Table III and are shown graphically in Fig. 1, in which also is shown the absolute resistivity of pure iron.

TABLE III.

Steel	Percent Carbon	Difference of Resistivity of Steel Tempered at 105° C. after Quenching and Same Steel Annealed Measured at					Increase in Resistivity of Solutes 25° C.-100° C.
		25° C.	40° C.	60° C.	80° C.	100° C.	
SN3	0.104	1.85	1.80	1.72	1.64	1.50	—0.35
M8	0.326	3.31	3.31	3.25	3.23	3.12	—0.19
H12	0.61	7.33	7.33	7.32	7.37	7.37	0.04
C5	0.945	9.19	9.44	9.62	9.72	9.77	0.58
SN1	1.11	21.97	22.09	22.24	22.38	22.33	0.36
MN1	1.15	14.23	14.54	14.85	15.01	15.01	0.78
INC	1.18	14.51	14.76	15.08	15.43	15.93	1.42

The results of the thermo-electromotive potential measurements are given in Table IV, in which the steels are arranged in the same order as in the preceding table and shown graphically in Fig. 2. In this table the thermo-electromotive potential of the freshly quenched and that of the samples tempered at 105° C., is the potential due to those solutes which are in solution in the quenched or tempered samples, but which are not in solution in the same steel in the annealed condition since in making these determinations the steel in the annealed condition is opposed to the same steel quenched or tempered, as the case may be. The thermo-electromotive potential of the annealed steels was the difference in the thermo-electromotive potential of the annealed steel and that of pure iron, since in making these determinations bars of the annealed steels were opposed to triple bars of ingot iron. The thermo-electromotive potential of quenched or tempered bars may be readily calculated by adding the thermo-electromotive potential obtained when these bars are opposed to the same steel annealed to that obtained when the annealed bars are opposed to ingot iron.

In Table V are given the thermo-electromotive potentials of three steels in both the annealed and tempered condition when opposed to pure iron. The reason for selecting these steels is

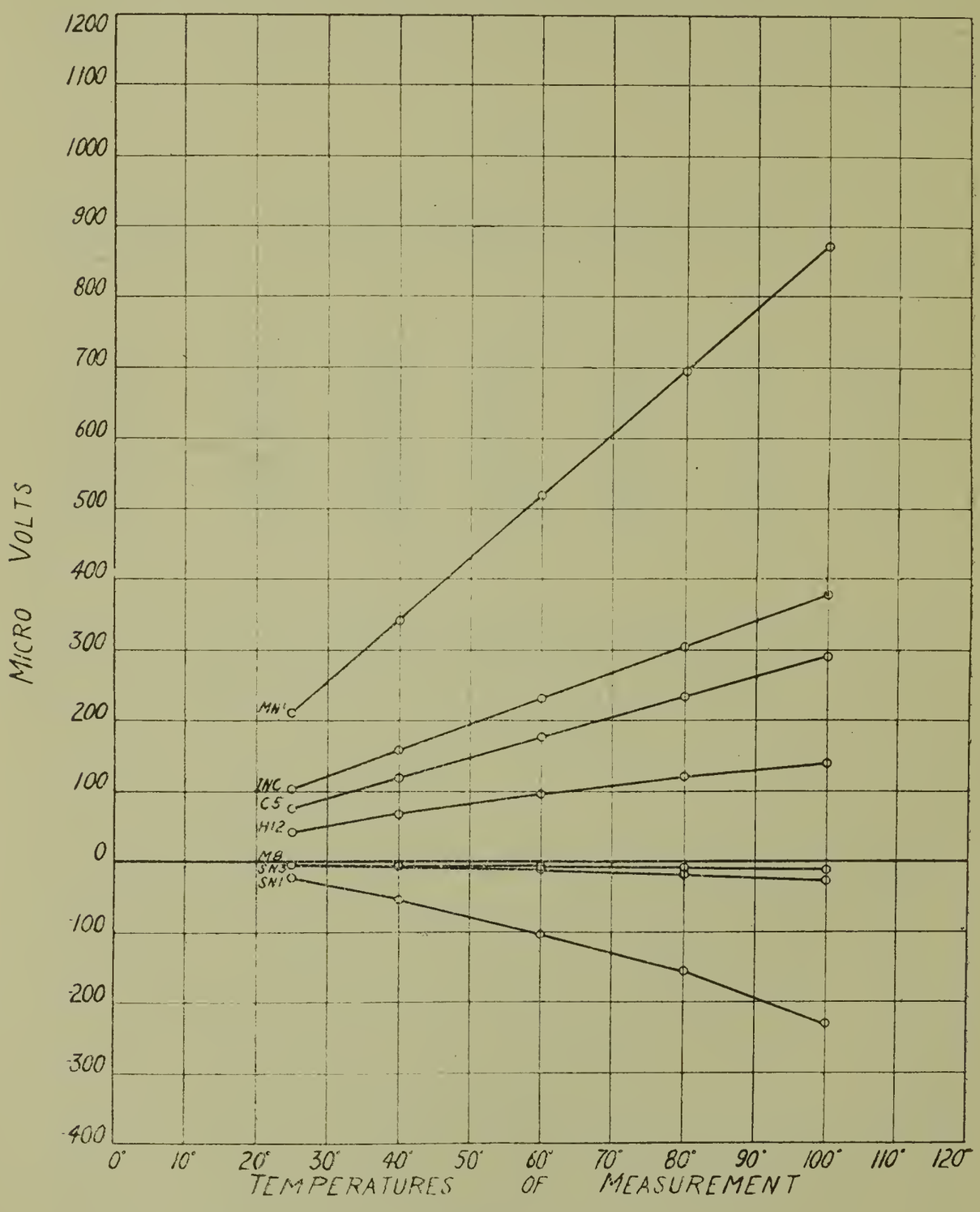
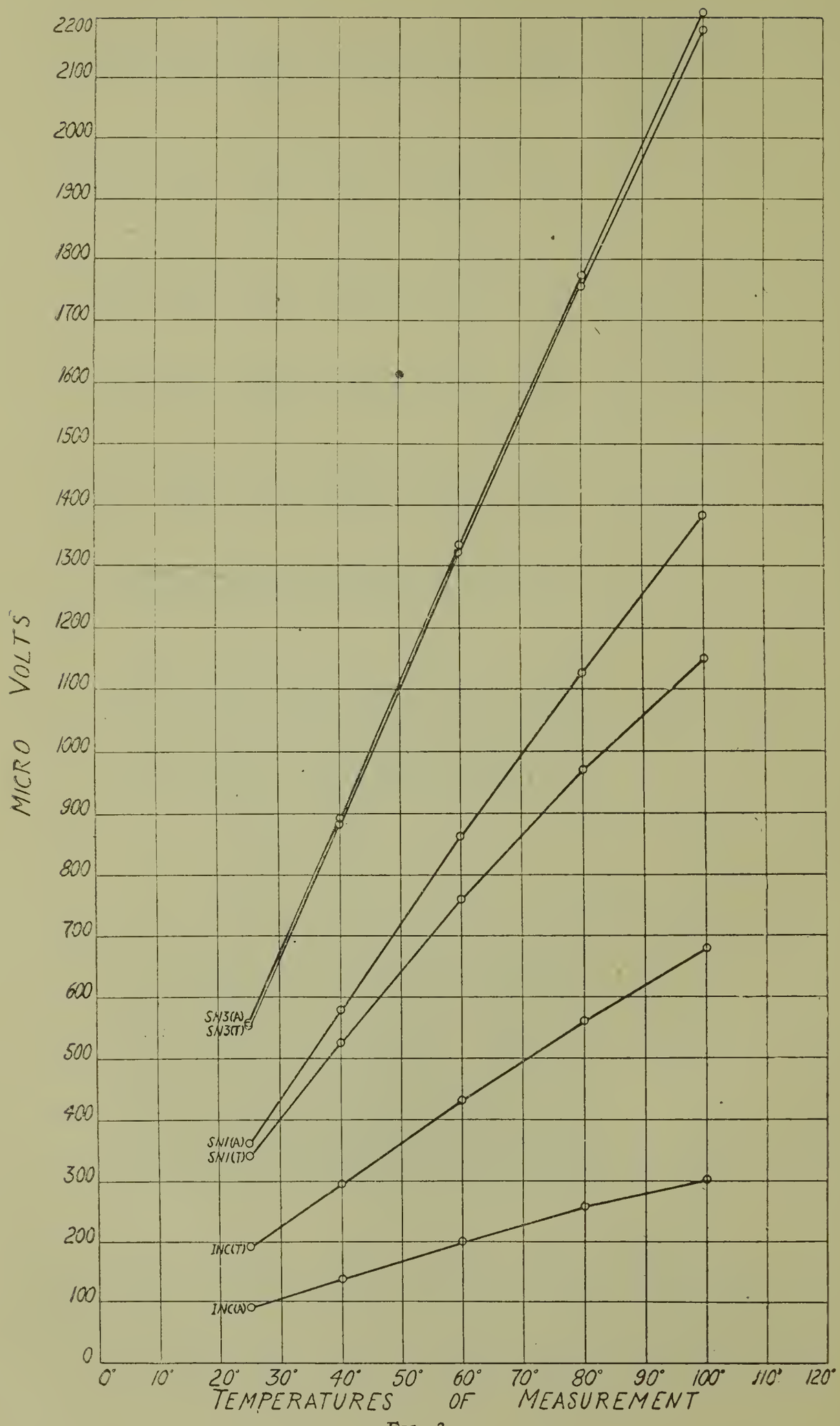


FIG. 2.

that in the steel marked INC we have a high concentration of carbides only as the solutes, in SN3 there is a very high concentration of silicides with a small content of carbides, and in SN1

TABLE IV.

Steel	Percent Carbon	Heat Treatment	Opposed to	Thermo E. M. F. Measured at					Increase in Th. E. M. F. of Solutes 25° C.-100° C.
				25° C.	40° C.	60° C.	80° C.	100° C.	
SN3	0.104	Quenched from 1002° C.	SN3 annealed	0					
		Quenched and tempered at 105° C.	SN3 annealed	—3	—6	—10	—16	—27	—24
		Annealed from 985° C.	Ingot Iron	556	890	1334	1772	2206	1650
M8	0.326	Quenched from 900° C.	M8 annealed	—3					
		Quenched and tempered at 105° C.	M8 annealed	—4	—5	—6	—8	—12	—8
		Annealed	Ingot Iron	371	596	894	1186	1468	1097
H12	0.61	Quenched from 900° C.	H12 annealed	80					
		Quenched and tempered at 105° C.	H12 annealed	44	68	96	120	138	94
		Annealed from 720° C.	Ingot Iron	138	218	320	412	490	352
C5	0.945	Quenched from 902° C.	C5 annealed	192					
		Quenched and tempered at 105° C.	C5 annealed	76	118	176	234	290	214
		Annealed from 903° C.	Ingot Iron	128	204	292	374	452	324
SN1	1.11	Quenched from 1002° C.	SN1 annealed	—4					
		Quenched and tempered at 105° C.	SN1 annealed	—22	—54	—102	—156	—232	—210
		Annealed from 950° C.	Ingot Iron	362	578	862	1126	1382	1020
MN1	1.15	Quenched from 1002° C.	MN1 annealed	216					
		Quenched and tempered at 105° C.	MN1 annealed	212	340	518	694	872	660
		Annealed from 810° C.	Ingot Iron	334	524	764	990	1210	876
INC	1.18	Quenched from 902° C.	INC annealed	262					
		Quenched and tempered at 105° C.	INC annealed	102	158	230	302	376	278
		Annealed from 905° C.	Ingot Iron	90	138	200	258	302	212


MICRO VOLTS
2200
2100
2000
1900
1800
1700
1600
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1400
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1200
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700
600
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400
300
200
100
0
SN3(A)
SN3(T)
SN1(A)
SN1(T)
INC(T)
INC(A)
0° 10° 20° 30° 40° 50° 60° 70° 80° 90° 100° 110° 120°
TEMPERATURES OF MEASUREMENT

FIG. 3.

TABLE V.

Steel	Percent Carbon	Heat Treatment	Opposed to	Thermo E. M. F. Measured at				
				25° C.	40° C.	60° C.	80° C.	100° C.
SN3	0.104	Annealed from 985° C.	Ingot Iron	556	890	1334	1772	2206
		Quenched and tempered at 105° C.	Ingot Iron	553	884	1324	1756	2179
SN1	1.11	Annealed from 950° C.	Ingot Iron	362	578	862	1126	1382
		Quenched and tempered at 105° C.	Ingot Iron	340	524	760	970	1150
INC	1.18	Annealed from 905° C.	Ingot Iron	90	138	200	258	302
		Quenched and tempered at 105° C.	Ingot Iron	192	296	430	560	678

there is a rather high concentration of both carbides and silicides. The results given in Table V are shown graphically in Fig. 3.

An examination of the data given in Tables II and III and of the curves shown in Fig. 1, clearly reveals that the portion of the electrical resistivity due to the carbides or other solutes in solution does not follow the same law, as far as the influence of temperature of measurement goes, as does the electrical resistivity of pure iron, the solvent. The electrical resistivity due to carbides increases somewhat with the temperature of measurement, although the coefficient of this increase would not be more than about one-fourth that of pure iron, while the electrical resistivity due to silicides in solution has a small negative temperature coefficient. The temperature coefficient of the electrical resistivity due to tungsten or nickel in solution seems to be also slightly negative.

Viewed in the light of the solution theory in the full sense of the term, the influence of heat treatment on the resistivity of steels becomes quite clear. If in any solution it is those molecules of solutes in solution which are most reactive toward different forms of energy, then a piece of steel in the freshly hardened condition should have its maximum specific resistance since such a piece of freshly hardened metal would have the maximum proportion of its solutes in solution and consequently these solutes would transform a maximum amount of electrical energy into heat, thus causing maximum electrical resistivity. The electrical resistivity of steel is a function of the total molecular concentration of all solutes actually in solution.

A study of the data in Tables IV and V and of the curves shown in Figs. 2 and 3 reveals at once that the thermo-electromotive potential due to solutes in solution is not a function of the molecular concentration of the solutes actually in solution, as is the electrical resistivity. From the data in Table IV and curves in Fig. 2 it will be noted from steels C5, INC and MN1 that, within limits of experimental error and over the temperature range 0° C. to 100° C., the thermo-electromotive potential due to a single solute in solution is a linear function of the temperature. In case of two or more solutes in solution at the same time this would not hold, as is shown in case of steels SN1, SN3, M8 and H12. The thermo-electromotive potential of a single solute is

dependent upon the composition of the solute and its concentration. A hypothesis that the thermo-electromotive potential of a solute in metallic solution is analogous to the solution tension of an electrolyte in an aqueous solution, would offer a reasonable explanation for the thermo-electromotive potential results found in the present investigation. This is best shown from the data in Table V and curves in Fig. 3.

If an electrolytic cell be set up with a single electrolyte A, the electromotive force of such cell will depend upon the solution tension of the electrolyte and the concentration. If a second cell with a single electrolyte B, whose solution tension is higher than that of A, be set up, it will also have an electromotive force dependent upon the solution tension and concentration of the electrolyte B. If a third cell be set up with a mixture of the electrolytes A and B, it will have an electromotive force intermediate between that of A and B, always being higher than that which would be found with A alone, but lower than if B were the only electrolyte. Electrical conductance in aqueous solutions is dependent upon the total ionic concentration, but is not dependent upon the solution tension of the solutes, this latter being wholly dependent upon chemical composition.

In steel INC we have a single solute, carbides, the thermo-electromotive potential of which is dependent upon the concentration of the carbides brought into solution by hardening. The thermo-electromotive potential of the hardened metal is higher than that of the same steel in the annealed condition. In case of the steel SN3 we have a very high concentration of silicides which seems to be almost as soluble in *alpha* iron as in iron in the *gamma* form; but in the annealed metal the carbides corresponding to 0.104 percent carbon would be almost completely precipitated. On hardening such a metal the carbides would go into solution and we would have in solution a high concentration of silicides together with a low concentration of carbides and, in consequence, a thermo-electromotive potential slightly lower than that which is given by the annealed steel in which the silicides alone were in solution. Owing, however, to a slight increase in molecular concentration of solutes in the hardened metal,

the electrical resistivity is correspondingly increased. This effect of two solutes in solution is shown still more strikingly in the case of SN1 in which we have 1.20 percent and 1.11 percent carbon. In the annealed condition the greater part of the carbides would be precipitated and the thermo-electromotive potential would be due to the silicides alone. On hardening, however, the carbides would pass into solution, and since the thermo-electromotive potential of carbides is much less than that of silicides, the thermo-electromotive potential of the hardened or tempered metal would be lower than that of the same metal annealed, although the specific resistance would be very much increased.

SUMMARY OF CONCLUSIONS.

1. The total electrical resistivity of steel is made up of two components, that portion due to the solvent iron, and that portion due to solutes in solution, the two portions not following the same laws so far as the temperature of measurement is concerned.

2. The temperature coefficient of the electrical resistivity due to carbides in solution is positive, but only about one-fourth as great as that of pure iron.

3. The temperature coefficient of the electrical resistivity due to silicides, as well as that portion due to tungsten and nickel in solution, is small but negative.

4. The thermo-electromotive potential of a single solute in solution is dependent upon the chemical composition and concentration and is a linear function of the temperature.

5. When two or more solutes are in solution at the same time the thermo-electromotive potential due to these solutes is not a linear function of the temperature and not dependent upon the total molecular concentration.

6. To account for the observed phenomena of thermo-electromotive potential, the hypothesis is advanced that the thermo-electromotive potential of solutes in metallic solution is analogous to the solution tension of electrolytes in aqueous solution.

University of Michigan,
June, 1917.

DISCUSSION.

PROF. W. D. BANCROFT[1]: This is a very interesting paper, but I think there are one or two points that the author either overlooked or does not lay quite sufficient stress upon. For instance, I do not believe that you can get anywhere, considering the resistance of the two-phase system, if you simply take into account the composition and ignore the structure. Suppose there are two phases, one of high and the other of low resistance. Now, if the phase having the high resistance happens to be the internal phase, and the other one is the external, or continuous phase, then the high resistance part merely acts more or less as a hole, as the current does not flow through it to any appreciable extent. If, on the other hand, you have the phase of low resistance, surrounded by the high resistance phase, the current must flow through the latter and you get an entirely different resistance.

This was brought out clearly by Mr. Fink in mixtures of thoria and tungsten. With the same percentage composition he got either a white mixture which did not conduct electricity or a black one which did, depending on the relative sizes of the two powders. If you have an interlacing system, where each phase is continuous, you get another state of things. I see no satisfactory way of accounting for the two-phase system if you ignore the structure, which Mr. Campbell seems to have done.

Then, as regards the thermo-electric force, that is not analogous to the potential difference between a metal and a solution. In the latter case you get transport of ponderable material when the current flows. If you have the solid metal in contact with a solid solution, you have no transport of ponderable material, and consequently the two things are quite different. The thermo-electric force is an effect due to difference of temperature, and it would correspond to the thermo-electric force of a Daniel cell, which depends upon the difference in the solution pressures of the two metals due to change of temperature, which is a different thing from what Mr. Campbell says.

So, while the data are of great interest and value, I am not so enthusiastic about the hypotheses which accompany them.

[1] Professor of Chemistry, Cornell University.

PRESIDENT C. G. FINK[2]: I should like to substantiate Dr. Bancroft's statement in so far as I disagree with Dr. Campbell. I do not see how Dr. Campbell's theory will account for conduction through a crystal, when its ohmic resistance is high, and that of the interface film is low. Why should the current prefer to go through the point of high resistance? A little of it will go that way, to be sure, but most of the current flow will be over the surface of the crystal.

In another case, we have a high resistance film, and a high conductivity crystal; in this case the current will puncture the film and pass through the crystal. (See Journ. Physical Chem., **21**, 32 (1917).)

J. W. RICHARDS[3]: I have prepared some notes on this paper, particularly from the electrical standpoint, and I beg your indulgence if I repeat some of the points which Dr. Bancroft and the Chairman have raised.

The experimental data which Prof. Campbell has determined and recorded in the tables I regard as valuable experimental data. I believe that is all of value to be found in the paper.

The conclusions drawn from the data are based on a theory which may or may not be true, and by means of inferences from the data, most of which are inadmissible. If we measure the resistivity of an iron alloy, it is not admissible to deduct the resistivity of pure iron from the observed resistivity. The difference does not mean anything whatever; above all, it is not and cannot be the resistivity of the other components of the steel. The principle of subtracting two resistivities, and then drawing conclusions as to the relative resistivities of the solute and that of the pure iron, gives no information of value, but figures which may be very misleading.

I therefore wish to place on record my opinion that while the experimental data given by Prof. Campbell are valuable, the conclusions drawn are entirely misleading.

C. W. SPICER[4] (*Communicated*): The information contained in this paper is closely related to a subject which I have had in mind for several years but which I have never demonstrated.

[2] Director of Research, Chile Exploration Company, New York City.
[3] Professor of Metallurgy, Lehigh University.
[4] Chief Engineer, Spicer Mfg. Co., Plainfield, N. J.

It is a well-known fact that the resistivity of steel varies with the carbon content. With this as a basis I have had in mind the possibility of roughly checking the uniformity of the carbon content of a shipment of billet-rolled steel bars as received from the mill. While these bars vary somewhat in size I have established the fact that the cross-sectional area varies only a small percentage as much as the variation in the electrical resistivity due to variation in carbon content, assuming that this latter variation is greater than the commercial limits specified for the steel.

It would, of course, not be possible by the electrical test to even approximately determine the actual carbon content. All that was intended to prove was that any given lot was reasonably uniform. It is a well-known fact that as steel is handled in the ordinary mill, a stray billet from some other heat will sometimes get into a lot of billets which are supposed to be entirely from one heat. This stray billet may be of steel of such a radically different nature from the lot into which it strays, that its undetected use might be a very serious matter. The chemist at the receiving plant might check the mill analysis by taking samples from several bars out of the shipment, and still not catch the stray bar.

The electrical test I have in mind would consist of two rugged contacts across which each bar could be laid, at some part of the process of unloading from the railway car and piling in the yard, and the resistivity measured by one of several possible ways. The process could be made extremely simple and rapid if the results could be relied upon, and would provide a quick, simple and reasonably positive method of checking every bar for uniformity with the rest of the lot.

The specifications of a large portion of our tonnage are as follows:

Carbon	0.35 to 0.45 percent
Manganese	0.50 to 0.80 percent
Sulphur	below 0.04 percent
Phosphorus	below 0.04 percent

The bars are squares and rounds, ranging from ¾ to 4 inch.

The data in Professor Campbell's paper, at least superficially, seem to indicate that the structural condition of the steel due to heat treatment has many times greater effect than the variation in carbon. If this is true, then the temperature at the final pas-

sage of the various bars through the rolls might vary so much in a given heat that such an electrical test as I have suggested would be of no value whatever. I would appreciate some comments by Prof. Campbell on this subject in the discussion of his paper.

PROF. E. D. CAMPBELL (*Communicated*): Although few chemists could probably be found who would consider a mixture of thoria and tungsten as constituting a two-phase system, yet both Dr. Bancroft and Dr. Fink apparently so consider it, and they both apparently hold that the resistivity in a solid solution is due to a purely mechanical obstruction to the passage of the current, and is not, as has been assumed by the author, dependent upon the chemical constitution of the solid solution, that is, on the atomic relation existing between the solutes and solvent which make up the solid solution. They assume that it is the physical structure, not the chemical constitution, on which the resistivity primarily depends.

In reply to their contention we would mention a few facts which may bear on this point. If a hypo-eutectoid carbon steel is cooled very slowly from a temperature of about 1,000° C., or above, the crystals or grains will be found quite large, and the precipitated carbides, constituting the cementite, will be found in comparatively large masses imbedded in the *alpha* iron which constitutes the main mass of the system. If another piece of the same steel be first hardened by quenching from a temperature of about 900° C., reheated to about 600° C., and then allowed to cool slowly, the precipitated carbides constituting the cementite will be found to be in the form of very fine particles, fairly uniformly distributed throughout the iron in which they are imbedded. The total mass of cementite and of imbedding iron would be substantially the same in both cases, but the size of the particles would be very different. If the resistivity is due to a purely mechanical obstruction, these two samples, treated as above, should have widely different resistivity, but, as a matter of fact, if there is any difference it is very slight.

Again, if a bar of ordinary hypo-eutectoid steel, air-cooled as it comes from the rolls, be cut into a number of pieces, one may be subjected to torsion through an angle of 40° to the inch; an-

other may be stretched cold until it has been elongated 15 percent; another may be compressed cold until it has been shortened 10 percent; still another may be cold hammered first in one direction and then in another until the grains are very much broken, and the Brinnel hardness number has been doubled or trebled; and yet the electrical resistivity of such cold-worked metal will be unchanged, or, if changed at all, the amount is so slight as to be negligible when compared with the changes induced by heat treatment, which will change the concentration of carbides in solution. The drastic cold working of the steel would very profoundly modify the size and shape of the crystals or grains. It would produce an enormous amount of surface film at the surfaces of the fractured grains, and would leave the metal in a state of strain. Many of the physical properties, particularly the elastic properties, as well as ductility and hardness, would be very much modified by the cold working; and yet this cold working, which would not change the chemical constitution, that is, the atomic relations existing between the solutes and the solvent, exerts very little, if any, influence on the electrical resistivity. If, then, the size of the crystals or grains, the size of the particles of solutes mechanically mixed with the solvent, the presence or absence of surface films, the existence or non-existence of a state of strain, has a negligible influence on the resistivity, by a process of exclusion, we are almost compelled to attribute that portion of the total resistivity not due to pure metal constituting the solvent, to the form in which the solutes are present.

Dr. Bancroft does not seem to have a very clear idea of the author's hypothesis to account for the thermo-electromotive potential of solutes in solution. In this hypothesis the author assumes that a given solute in solid solution possesses, as does an electrolyte in an aqueous solution, a certain solution tension which is dependent upon the chemical composition of the solute and the temperature. If, then, under this conception, there is a difference of temperature at two points of a metallic solution there will be a difference in thermo-electromotive potential at these two points, which may be measured by means of a potentiometer or other suitable apparatus.

The author is gratified that Dr. J. W. Richards admits the value of the experimental data given in the tables, and has not

utterly condemned the entire paper. In much of the research work carried on by Le Chatelier, Benedicks, and numerous other experimenters during the past quarter century, the necessity of differentiating between that portion of the total resistivity due to the solvent and that portion due to the solutes is clearly recognized. So that unless Dr. Richards can suggest some better method for differentiating between these two than the one commonly employed by experimenters in this field, the author will feel justified in employing the long-recognized method in spite of Dr. Richards' dictum as to its inadmissibility.

In reply to Mr. Spicer's inquiry concerning the feasibility of a method for checking up the uniformity of a lot of steel bars by measuring the resistivity of each bar, the author feels that although such a process seems at first glance to be very attractive, Mr. Spicer's conclusions, as indicated in the last paragraph of his discussion, are well founded. The only case with which the author is familiar in which the electrical resistivity of steel is used as a criterion of its fitness is in the manufacture of conductivity steel to be used in the third-rail system of electric roads. Experience in the manufacture of this class of steel has demonstrated the necessity of making the metal as nearly pure iron as can be produced consistent with good rolling qualities.

Ann Arbor, Mich.
Jan. 3, 1918.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

CALORIZED IRON AS AN ELEMENT FOR THERMOCOUPLES

By O. L. Kowalke.*

[*Abstract.*]

The author compares the thermo-electromotive force of couples of "calorized" iron-constantan with that of "uncalorized" iron-constantan. A sample of "calorized" iron wire from the General Electric Company gave the same thermo-electric potentials as "uncalorized" iron, up to 1,000° C., on repeated calibrations. A sample from the Brown Instrument Company gave slightly different potentials until after it had been heat treated at 800° C., after which it gave similar results to the other "calorized" wire. The author concludes that a heavy wire, lightly calorized, gives as constant results as uncalorized iron, and has a much longer life.

There are in the market several thermocouples, having iron as one of the elements, which have high electromotive forces and are fairly constant, but have the disadvantage that the iron element oxidizes rapidly above 500° C. If iron could be protected from oxidation by some sort of a coating which would not greatly change the electromotive force, its usefulness as an element for thermocouples would be much extended.

It has been shown by W. E. Ruder[1] that "calorizing" iron, "which consists in producing a rich aluminum alloy upon the surface of the metal," practically prevents oxidation below 1,000° C. It was therefore of interest to determine whether "calorized" iron wire was suitable for thermocouples and whether its electromotive force was as high and as constant as the uncalorized wire. One piece of "calorized" iron wire was donated by the Research Laboratory, General Electric Company, through the courtesy of Mr. F. W. Gillette, of the Diamond Power Specialty Company;

* Professor of Chemical Engineering, University of Wisconsin.

[1] Trans. Am. Electrochem. Soc., **27**, 253 (1915).

another piece of "calorized" iron wire was purchased from the Brown Instrument Company. The wire from the General Electric Company was 0.174 inch (0.44 c.m.) diameter and straight; the wire from the Brown Instrument Company was 0.137 inch (0.34 c.m.) diameter and was bent double on itself so that in straightening it out some of the alloy chipped off.

Grateful acknowledgment is hereby made to the General Electric Company for donating the wire and to Mr. M. A. Powers for assistance in obtaining these data.

Couples Used: Three sets of couples were made, each being 12 inches (30.5 c.m.) long. The wires in each couple were fused together at one end in an electric arc after filing off the "calorized" coating at the point of fusion. The wires were insulated from one another by porcelain bushings, and flexible copper lamp cord was soldered to the elements for leads. The wires used in all the couples were annealed by heating to a bright red with electric current.

Constantan wire, purchased from Leeds & Northrup, was used in all the couples as one element. The combinations used in these tests were:

Couple No. *10B* =	constantan	*vs.* "calorized" iron	(Gen. Elec. Co.)
" " *32B* =	"	*vs.* " "	(Brown Instr. Co.)
" " *81B* =	"	*vs.* "uncalorized" iron	(Leeds & Northrup)

At the conclusion of the tests on the above couples, about 1½ inches (3.8 c.m.) was cut from the fused ends of the couples and remaining long portions fused together again. The couples so made were designated by the letter "C." Thus

Couple	*10C*	was	made	from	Couple	*10B*
"	*32C*	"	"	"	"	*32B*
"	*81C*	"	"	"	"	*81B*

Equipment Used: All electromotive force measurements were made on Leeds & Northrup portable potentiometers. A platinum vs. platinum-10 percent rhodium thermocouple calibrated by the Reichsanstalt in Berlin served as a standard for comparison. An electrically heated tube furnace, such as used in previous tests,[2] served for making the calibrations.

Procedure: Discs of asbestos were drawn over each couple to keep it in the axis of the furnace, and the ends of the furnaces

[2] Trans. Am. Electrochem. Soc., **29**, 561 (1916).

were closed with loose magnesia-asbestos. The couples were all inserted to a length of about 5 inches (12.7 c.m.) into the furnace for all calibrations and heat treatments.

In all tests only one couple at a time was compared with the standard platinum couple. The temperature intervals of the points of comparison were about 100° C., and at each point the temperature was maintained constant for a period of two minutes to insure equilibrium. The couples were calibrated as annealed, then they were heated in an electric tube furnace for 24 hours at 800° C., and given a second calibration, then heated again for 24 hours at 900° C. and calibrated a third time.

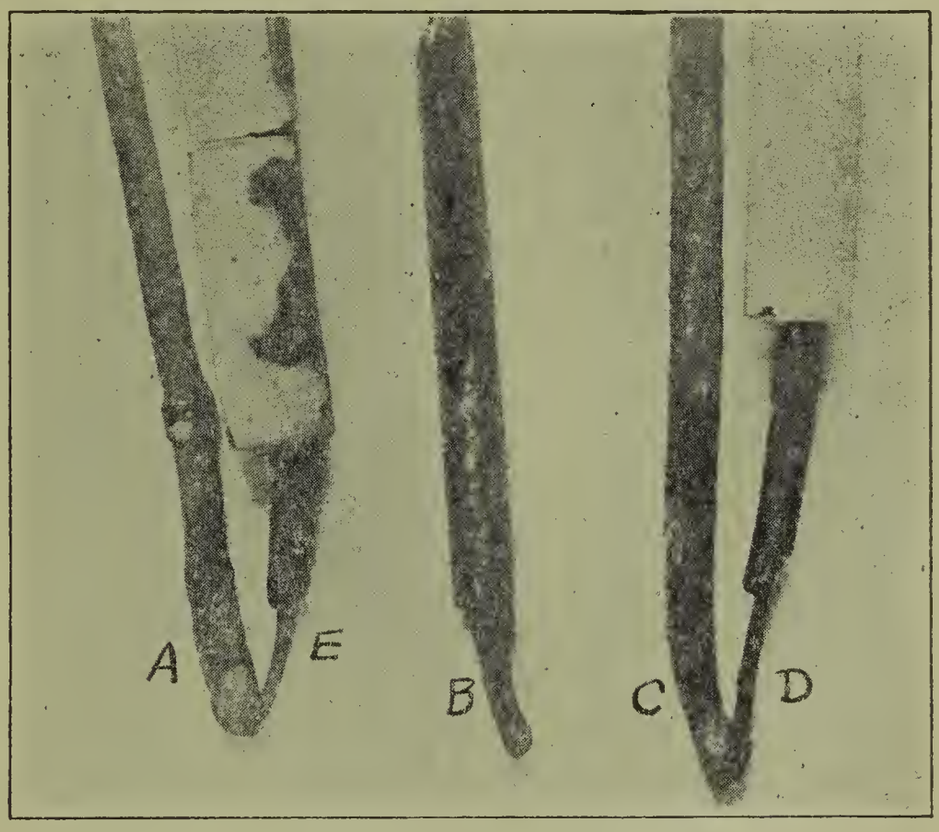


FIG. 1.

After the third calibration about 1½ inches (3.8 c.m.) was cut from the hot junction end of each couple and the remainder fused together again. The remaining couple was called *C*, and was calibrated only at three points to see whether any change in calibration had taken place due to changes in the wires or method of fusion.

Results and Observations: As shown in Fig. 1 above, the constantan wires *E* and *D* and the uncalorized iron wire *B* have oxidized badly. The "calorized" wires *A* from Brown Instrument

Company (couple *32B*), and *C* from General Electric Company (couple *10B*) have not oxidized to any extent as a result of three calibrations and two heat treatments.

On the General Electric Company wire *C*, Fig. 1, there were narrow ridges of iron oxide running longitudinally with the wire. On the whole, the wire was in good condition at the end of the tests.

The wire from the Brown Instrument Company, *A*, Fig. 1, had spots of iron oxide in various places where the wire had been bent and the aluminum alloy chipped off. Owing to the chipping the wire *A* was not in such good condition as wire *B*.

A microscopic examination of the wires as received showed that the coating on the General Electric Company wire was very much thinner than that on the Brown Instrument Company wire. As a result of prolonged heating, the coating of iron aluminum alloy penetrated into the wire. In the wire from the Brown Instrument Company the alloy had penetrated about one-half the radius of the wire; in the wire of the General Electric Co. the alloy did not penetrate so deeply. The observations on microstructure were identical with those of W. E. Ruder.[3]

In Fig. 2 is shown the comparison of the first calibrations of all the samples. It will be noted that couple *32B* on the first calibration gives a higher electromotive force for a given temperature than the other two couples. On repeated calibrations, the latter couples also gave concordant results, but *32B* discordant results. After heat treatment of *32B* at 800° C., however, its calibrations became concordant, and the same as the other couples, showing that it was probably not sufficiently annealed at the beginning.

These tests have not, of course, shown what would happen when the core of iron was all changed to the alloy of iron and aluminum. Before such thing would take place the companion wire, constantan, would be used up.

CONCLUSIONS.

1. "Calorized" iron may be a suitable material to replace iron in the iron-constantan combination, because it has the same electromotive force and constancy as uncalorized iron, but has a much longer life.

[3] *Loc. cit.*

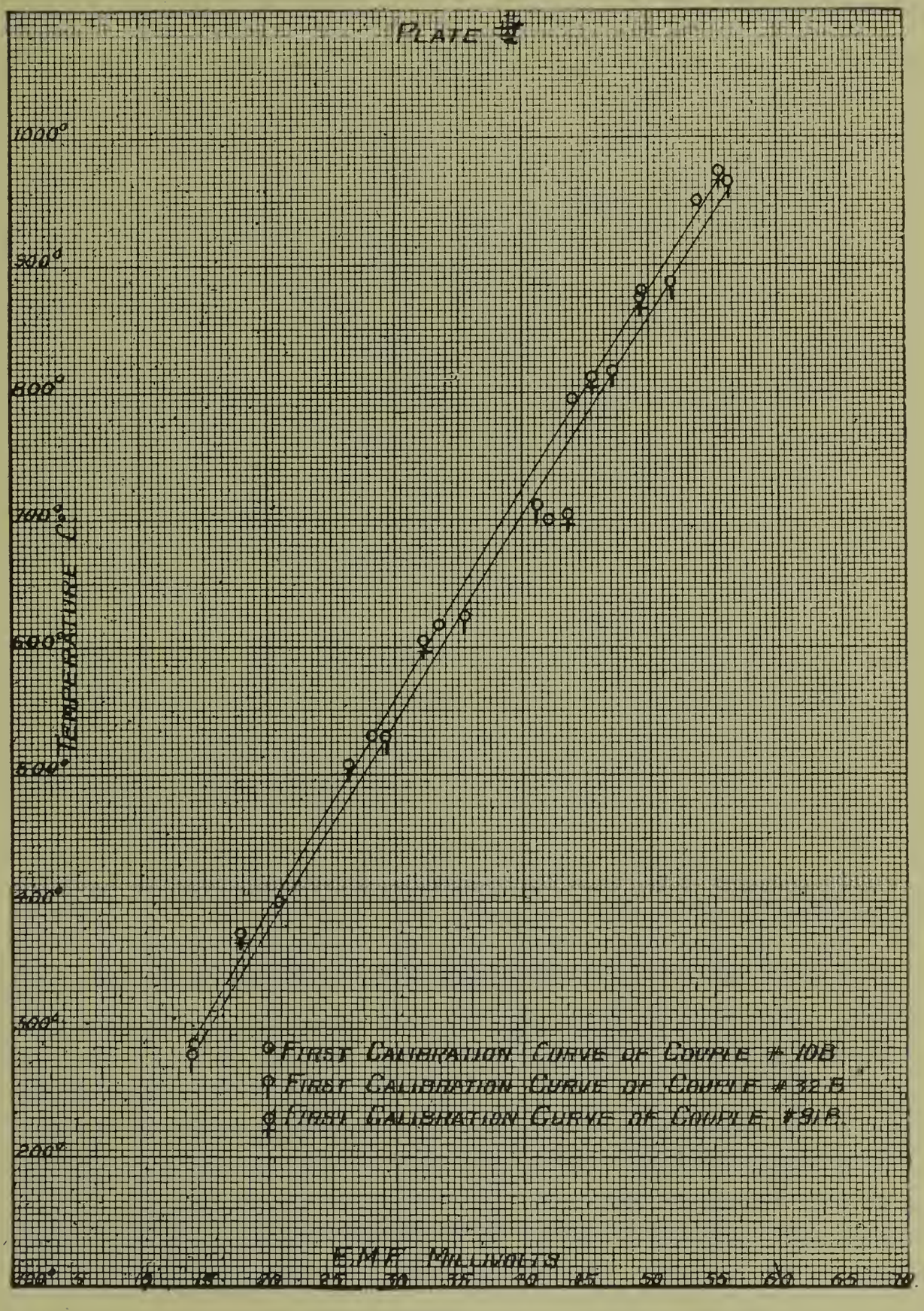


FIG. 2.

2. A thin coating of "calorizing" is effective, and is to be preferred over a thick coating because of the greater liability for a thick coating to flake off on bending.

3. The "calorized" wire should be a fairly heavy one, to avoid bending and to provide a sufficiently large iron core inside the iron-aluminum coating.

Chemical Engineering Laboratories,
University of Wisconsin.

DISCUSSION.

MR. W. RUDER[1]: We are indebted to Mr. Kowalke for the results given to us in his paper, showing that the calorized iron gives a constant value of thermo-electric forces. The only criticism I can make is that Mr. Kowalke did not calorize both the constantan wire as well as the iron wires of his couple. There is no reason why that should not have been calorized as well as the iron.

Another disadvantage of calorizing the iron only is that the fusion of the end with the constantan would tend to diffuse the alloy on the surface and make it less protective.

If the thermo-couple were made up and then calorized, I think the results obtained would show much longer life, and that it could be used at a higher temperature than the ordinary base metal couple. We have calorized such couples, but I have no data to give on their thermo-electric values.

O. L. KOWALKE (*Communicated*): It would be of considerable assistance to investigators if information were available where calorizing could be obtained commercially.[2] Attempts to get the constantan wire calorized were not successful. No attempt to calorize the wire was made in this laboratory, since only commercial products were desired.

[1] General Electric Co., Schenectady.

[2] The Diamond Power Specialty Company, Detroit, Mich., does commercial calorizing.—ED.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 4, 1917, President Fink in the Chair.

COPPER CASTINGS FOR ELECTRICAL PURPOSES

By G. F. Comstock.*

[Abstract.]

The difficulty of obtaining sound castings of pure copper is ascribed by the author to its rapid absorption of oxygen and other gases when it is melted, the gases being evolved from the cooling copper, forming blow-holes, and the oxide forming films of copper oxide eutectic between the grains of metal.

After reviewing the history of attempts to avoid these difficulties, the results of using silicon as a deoxidizer in a certain manner are described, by which complicated and large sand castings are made with 75 to 85 percent electrical conductivity.

Photomicrographs of the castings cast without additions, badly oxidized, and deoxidized by various additions, are shown.

It is only within the last fifty years that the refining of copper has become so efficient as to make possible the transmission of power by electricity with negligible losses due to resistance. Before 1860 the resistance of the usual grades of copper, even in the form of wire, was only about 50 percent of the standard which we have today. Then Matthiessen brought out his classic work on the electrical conductivity of alloys, including the paper "On the Effect of the Presence of the Metals and Metalloids Upon the Electric Conducting Power of Pure Copper," published in "Philosophical Transactions," 1860, pp. 85 to 92. This work by Matthiessen emphasized the importance of eliminating all possible traces of impurities from copper to be used for conducting electricity. For instance, he found the conductivity of copper with 1.6 percent zinc to be only about 79 percent of that of pure copper,

* Metallurgist, The Titanium Alloy Mfg. Co., Niagara Falls, N. Y.

both being in the form of wire; with 1.3 percent tin it was decreased to about 50 percent, with 0.1 percent aluminum to 76.5 percent, with 0.5 percent iron to 36 percent, while only 0.13 percent phosphorus decreased the conductivity to 70 percent, and even traces of arsenic, to 60 percent.

The publication of these results had considerable influence in encouraging progress in the direction of more complete purification of copper in refineries, and soon the conductivity of the average copper wire turned out for electrical uses rose from around 50 percent to about 98 percent of the figure which copper should attain. At the present time it is unusual for standard copper wires to run much below 100 percent in conductivity, this figure meaning that a wire one meter long and of such diameter as to weigh one gram has a resistance of 0.15328 ohm at 20° C.

Before passing to the consideration of castings, which really form the subject of this paper, it is desired to make a brief reference to the work of Lawrence Addicks on the conductivity of various grades of copper. A paper by him was published in the Transactions of the American Institute of Mining Engineers, Vol. XXXVI (1906), page 18, on "The Effect of Impurities on the Electrical Conductivity of Commercial Copper," in which he gives the relative decrease in conductivity per unit of impurity added for sixteen of the more common impurities in copper wire. This factor of decrease in conductivity per unit addition is thus 720 for arsenic, 600 for phosphorus, 500 for aluminum, 140 for iron, 70 for silicon, 67 for tin, 30 for zinc, 25 for oxygen and 3 for lead. The above ratios are intended to apply only to small decreases in conductivity, or to very small additions of the respective elements.

Since it is known that in order to get copper wires of high conductivity it is necessary only to start with extremely pure copper bars, it might seem a simple matter to secure all kinds of copper castings of equally good conductivity by the same means, or merely by using pure copper. But two difficulties are encountered when this is attempted; first, sound castings are almost an impossibility, and second, the pure metal in the cast condition is too soft and weak to give the best satisfaction in most electrical uses.

The difficulty of obtaining sound castings of pure copper is caused by its rapid absorption of oxygen and other gases when

it is melted. The gases not only dissolve in the molten metal but copper oxide is also formed, which is held in solution until the metal freezes. When a casting is poured, the gases are evolved from the cooling copper forming blow-holes, and the oxide comes out of solution forming films of a copper-oxide eutectic between the grains of metal. These films and gas holes naturally impair the electrical conductivity of a casting as well as weaken it mechanically, because of the presence of less pure copper in a given cross-section. Casting in a cold metal mold, which chills the copper very fast, reduces the severity of the unsoundness due to gas holes, but most castings by reason of complicated form must be cast in sand to make their production financially practicable.

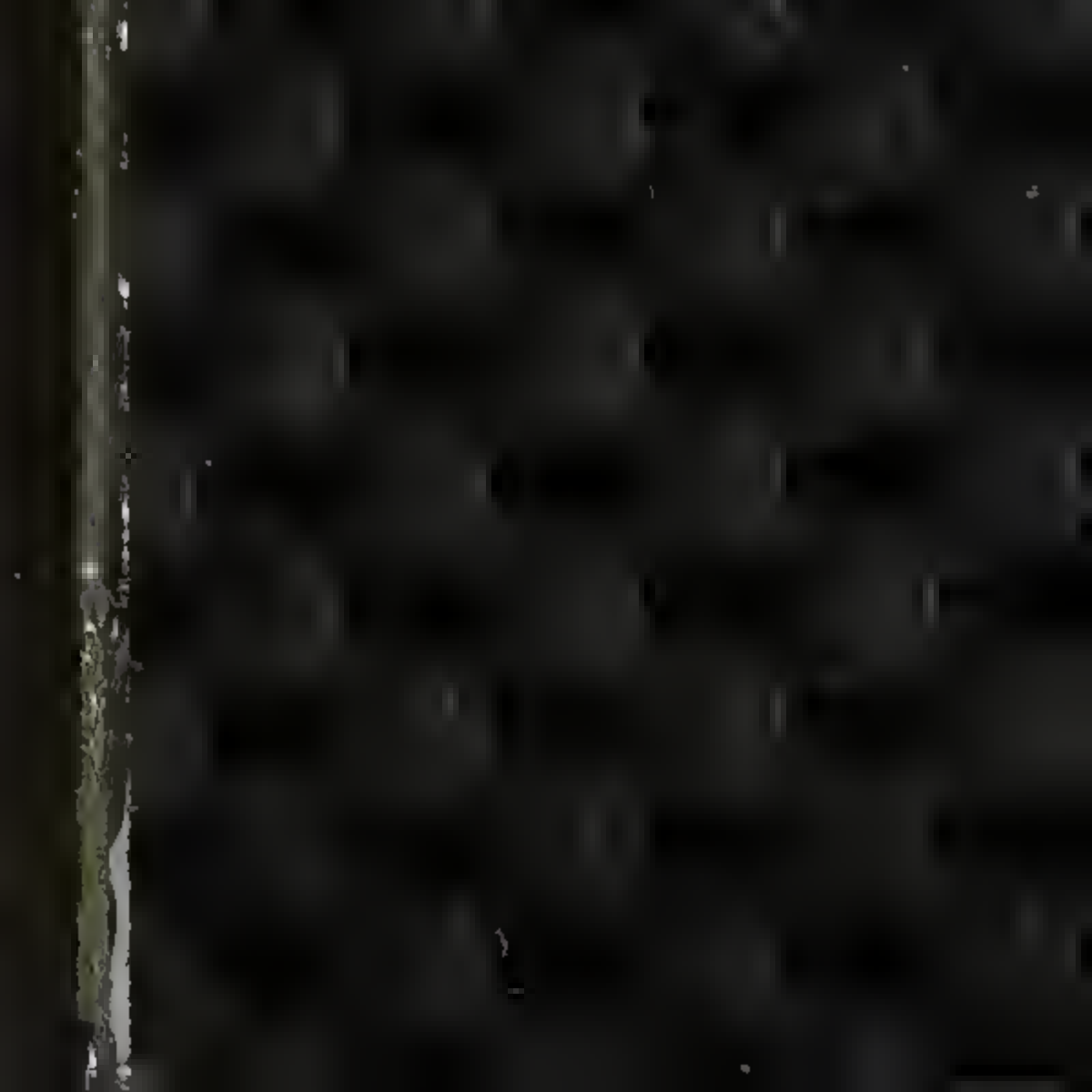
The second difficulty with pure copper castings, that of softness and weakness, cannot be overcome except by adding some other metal to the copper, thus making it no longer pure, or by giving the casting some mechanical treatment such as forging, after which it is, properly speaking, no longer a casting. Where cast copper has strength of only 20,000 lb. per sq. in. (14 kg. per sq. mm.) and a Brinell hardness of about 35, small bars of the same material after forging may have a strength of 35,000 lb. per sq. in. (24.5 kg. per sq. mm.) and a Brinell hardness of 70.

Foundrymen called upon to make castings of copper for electrical uses have in the past generally added an impurity intentionally for the purpose of deoxidation, and have thus raised the hardness and strength of their castings, although at the expense of more or less conductivity. Zinc was the element most often used, and with its aid sound castings could be produced fairly easily with the minimum reduction of conductivity (as can be seen by referring to Matthiessen's and Addicks' data for wires quoted above). Of course, such castings deoxidized by zinc had a considerably lower conductivity than copper wire, running only about 40 to 50 percent in many cases. The necessity of sacrificing conductivity for the sake of soundness in the case of copper castings seems to have been recognized by electrical engineers, who designed the cast connections of important conductors with generous cross-sections to prevent heating. A better deoxidizer, which would give the same soundness obtained by zinc but without reducing the conductivity so much, is of course much to be desired, as it would allow a casting of less weight to carry with equal

ease any given amount of current. To this end phosphorus and silicon have both been used in place of zinc, but while very small quantities of either are normally sufficient, their use is attended by the disadvantage that an excess of either of these elments does more harm to the conductivity than an equal excess of zinc. This objection applies with more force to phosphorus than to silicon. Boron has lately been used with success as a deoxidizer for copper, but its use is attended with considerable expense because of the difficulty of reducing this element to a form in which it is effective.

The problem of a superior deoxidizer for copper has received serious study in the experimental department of the Titanium Alloy Mfg. Co. for several years, with the result that a method of using silicon as a deoxidizer for copper has been found which makes the most of the superiority of this element over zinc, and at the same time minimizes the bad effect of the small excess of deoxidizer which cannot always be avoided. Of course careful melting of the copper, to prevent as much oxidation or absorption of gas as possible, is an important part of this process. The first step, then, is to melt the metal quickly and in as non-oxidizing an atmosphere as possible, keeping it well covered with charcoal at all times. A small amount of silicon is added before pouring, in the form of a flux which we have developed and which acts in such a way that the excess of silicon and oxides of silicon are thrown out of the metal to the surface of the casting, leaving the interior pure and clean. In this way we have been able to make complicated and large sand-castings of pure copper having 75 to 85 percent conductivity, as compared with the 40 to 50 percent generally obtained on copper castings deoxidized by zinc. This material naturally is soft and weak, and so far we have found no way to harden it except by forging. But we are still working along these lines, and hope eventually to evolve an alloy which will make harder, stronger castings of equally good conductivity.

A few photomicrographs are submitted herewith, to illustrate the structures of a few samples of copper made in different ways. These specimens were examined in connection with the research to develop a satisfactory deoxidizer for copper. Fig. 1 shows the structure of a small ingot of especially pure electrolytic cop-



per magnified 100 diameters. A section of the metal was carefully polished to remove all but the finest scratches from the surface, which was then treated with a mixture of strong ammonia and hydrogen peroxide until is was rather deeply corroded. This etching reagent attacks the copper crystals with different intensities, so that some appear darker than others after etching. Impurities are shown either by dark streaks between the grains or by more or less regular markings within the grains. In this case there are no markings within the crystals, showing the absence of dissolved impurities, but there are dark streaks between the copper grains due to the presence of a little copper oxide. Fig. 2 shows a sample of copper which was carelessly melted and badly oxidized. An excessive amount of oxide is here indicated by the dark bands between and within the grains of pure copper. Fig. 3 is a view taken at a higher magnification (400 diameters), and before etching, of copper with abundance of oxide. The drop-like particles are blue copper-oxide, and the background in which they appear is pure copper. The clear areas of this view are thus the copper grains, and the spotted areas are what is known as a eutectic, or mixture, of copper and copper-oxide. It is this eutectic which is strongly attacked in etching, giving dark bands around the copper grains, and it is evident from this view that a very small amount of oxygen, present as tiny globules of Cu_2O, will form an appreciable amount of the eutectic, as was the case in the pure copper shown in Fig. 1.

Fig. 4 shows a sample of copper treated with phosphorus, etched as explained above, and magnified 100 diameters. The copper grains are seen to have etched darked around their edges, showing the presence of phosphorus in solution, and there are streaks of oxides between the grains. Fig. 5 shows some copper deoxidized and hardened by about 4 percent of zinc. This metal is sound and free from dirt or oxides, but the crystals show rows of light spots indicating an impurity (zinc) in solution. This view was magnified only 20 diameters.

Fig. 6, also magnified 20 diameters, shows some very impure cast copper, with traces of lead, tin and iron, as well as considerable zinc, and the unevenness of structure is very evident. Fig. 7 shows a similar impure sample containing a little arsenic, but magnified 100 diameters instead of 20.

Fig. 8 shows the structure of copper deoxidized with an excess of silicon, magnified 50 diameters. The patterns in the crystals are very distinct here, but the metal is sound and free from oxides. Fig. 9 shows a sample of the purest cast copper which we have been able to make, which is sound and clean and runs from 75 to 85 percent in conductivity. Only very few traces of oxides are present here in the form of black specks, and the purity of the copper is shown by the freedom from any light and dark patterns within the crystals. Fig. 10 shows this same copper after being hardened by forging, the crystals having been broken up into finer grains, but without oxidation or loss of purity or conductivity. All the views except Fig. 10 were of cast copper, and all except Fig. 3 show copper etched with ammonia and hydrogen peroxide. All except Nos. 3, 5, 6 and 8 were taken at a magnification of 100 diameters.

Niagara Falls, N. Y.
Sept. 29, 1917.

DISCUSSION.

PRESIDENT C. G. FINK[1]: Mr. Barre, how do the results obtained by silicon compare with boron?

H. A. BARRE: The conductivity is much higher than those figures. I do not remember the figures, but I think it is 85 to 86, and as high as 90 has readily been obtained.

J. W. RICHARDS[2]: By the use of boron or boron sub-oxide?

H. A. BARRE: By boron sub-oxide. Boron carbide is being put out now in this.

J. W. RICHARDS: The use of silicon as a deoxidizer of copper is quite old, and if there is any new specific way of using it, the paper does not describe it, so that it adds nothing to our knowledge of how to use silicon.

GENRY RANDALL: We have made extensive experiments with the use of magnesium as a deoxidizer in copper and brass cast-

[1] Director of Research, Chile Exploration Co., New York City.
[2] Professor of Metallurgy, Lehigh University.

ings. Between 0.2 percent and 0.3 percent added makes a very dense casting well adapted for castings for electric furnace work where high electrical conductivity is required.

D. F. Comstock (*Communicated*): The figures given by Mr. Barre do not seem to substantiate his statement that boron used as a deoxidizer for copper gives "much higher" conductivity than that obtained by us with silicon. We obtain regularly from 75 to 85 percent conductivity in our castings, and Mr. Barre's "85 to 86" cannot be said to be very much higher. Moreover, the figure 90 which he mentions has been matched by us with a result of 89.8 percent obtained at one of the U. S. Bureau of Standards laboratories. We admit that boron may give slightly better results than our flux, but not much better, and the expense of using it is decidedly greater.

Prof. Richards is doubtless correct in his remarks, and the writer must plead guilty to the charge of having added nothing to the knowledge of such eminent scientists as he.

In regard to magnesium as a deoxidizer for copper and brass, we have not had much success with it along just those lines, and feel that our silicon flux is superior for copper. But magnesium is nevertheless a powerful deoxidizer, and it would be far from correct to claim that it is was without value in non-ferrous alloys.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

ELECTROLYTIC RECOVERY OF COPPER AND SULPHURIC ACID FROM COPPER MILL PICKLING SOLUTIONS

By J. L. Jones.

[Abstract.]

Mention is made of precipitating copper from waste copper sulphate pickling solutions by iron scrap, also of neutralizing the excess of acid and evaporating down to copper sulphate crystals (bluestone). There is then described an electrolytic plant for extracting the copper in a pure condition and regenerating the acid for re-use. The acid content is run up to 20 percent and the copper content reduced to about 1 percent. Some of the copper is deposited directly on steel plates as copper plating, but most of it is collected as cathodes for re-melting.

In the rolling of copper it is necessary to remove the copper scale that is formed in the various heating and annealing operations. If this is not done the scale is rolled into the copper, giving a rough surface. Another reason for removing the scale is the fact that it is an abrasive, and where copper is drawn into bars, strap, or wire, the dies would soon be ruined if the scale were not removed.

The pickling of copper is done in large, lead-lined wooden tanks, using a solution of dilute sulphuric acid which is heated by waste steam. When the rate of pickling becomes slow, due to the solution becoming saturated with copper sulphate, more acid is added, and the tanks are often run until supersaturated. It is not an uncommon thing in some plants to find the pickling tanks a solid mass of copper sulphate crystals on Monday morning, if the steam has been turned off on Saturday and the pickling solution allowed to cool.

TREATMENT OF THE SOLUTION.

Two methods have generally been followed by manufacturers for utilizing the waste pickle after it has ceased to be satisfactory for cleaning copper, and a third has now been devised and put into operation.

(1) Precipitation of the Copper by Iron.

The spent acid is removed from the pickling tanks to suitable precipitating tanks and a previously calculated amount of sheet

FIG. 1.

steel or steel turnings is added. This addition serves to neutralize any free acid present and throws down metallic copper from the solution, with the formation of sulphate of iron. The sulphate of iron solution is allowed to run off, and the copper mud, without any washing or other treatment, is shoveled out, dried, and sent to the copper smelter.

This method of procedure has a number of disadvantages. While the copper is recovered it is not in its most valuable form.

The acid is not recovered, about 2 pounds of acid being lost for each pound of copper recovered. When soft steel or iron scrap is used for precipitating the copper, it is not uncommon to find pieces practically unacted upon at the end of six months, only the outside being coated with copper. By using cast iron borings and having an excess of acid the reduction is rapid, but the graphite, silicon, etc., from the cast iron renders the copper mud less valuable. The drying of the copper mud, freight to the smelter and the smelting charges, reduce the returns considerably.

(2) Manufacture of Bluestone Crystals.

Probably the most generally used method of utilizing the spent acid from the pickling of copper is to convert it into sulphate of copper or bluestone crystals. The spent pickle is neutralized as completely as possible by adding to it scrap copper or copper scale. It is next filtered and then concentrated by being allowed to evaporate spontaneously or by heating with a steam coil placed in the bottom of the tank. When the saturated solution cools the copper sulphate crystals are allowed to form on lead or copper plates suspended in the solution and also upon the sides of the tanks. The large blue crystals are quite beautiful if seen when first withdrawn from the mother liquor of the crystallizing tanks. Recrystallization gives a purer grade of sulphate of copper.

A process for making copper sulphate thus on a large scale has been patented by F. H. Daniels, A. F. Backlin and H. K. Swinscoe. Vacuum evaporators made of copper are used for the concentrating, and a centrifuge for removing any liquor that adheres to the crystals.

This method has the advantage over the precipitation by iron that it utilizes both the copper and the sulphuric acid. It has, however, certain disadvantages. The sulphate of copper must not only be manufactured but also packed and sold. A considerable amount of it is used in making up various sprays to destroy insects. The vine-growing districts of Austria, Italy, and other European countries imported large amounts before the war, but this outlet has been shut off, materially reducing the demand.

The strongest argument, however, against both of the above methods of handling waste pickling liquor is that the pickling

bath is, in use, constantly changing in acid content, and for this reason cannot be relied upon to do rapid and uniform work.

(3) Electrolytic Recovery.

In order to avoid the difficulties mentioned, and more especially to speed up production and secure a pickling bath that would do rapid and uniform work, the Westinghouse Electric and Manufacturing Company has installed in its new copper mill an electrolytic plant that recovers both the metallic copper from the waste pickle and also the sulphuric acid. Fig. 2 gives a perspective diagram of the entire recovery system, showing both the pickling and electrolytic tanks.

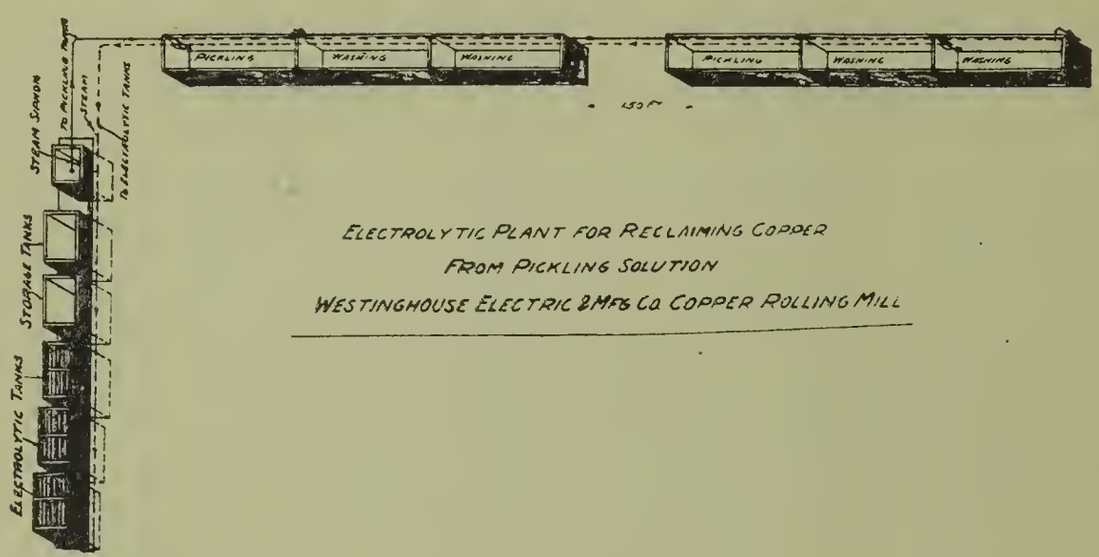


FIG. 2.

The pickling is done in two lead-lined wooden tanks, each 16 feet long, 4 feet wide and 3 feet deep (4.8 x 1.6 x 0.9 m.). They hold 1,300 gallons each (4,920 liters). A few gallons per minute of the pickling acid is allowed to flow by gravity from the pickling tanks and is lifted by a steam siphon to the first one of the series of recovery tanks. (See Fig. 3).

The recovery tanks are five in number. They are each 6 feet long, 3½ feet wide and 4½ feet deep (4.8 x 1.1 x 1.35 m.). They hold 535 gallons (2,000 liters) each and are lined with 7 lb. sheet lead (350 g./sq. dm.). Another smaller tank 4 feet long, 3 feet wide and 4½ feet deep (1.6 x 1.2 x 1.35 m.), holding 220 gallons (800 liters), serves as a storage tank or sump. The sump receives from the last recovery tank an amount of regenerated acid equiva-

lent to the amount of pickling acid received by the first recovery tank. The regenerated acid in the sump is returned by a steam siphon to the pickling tanks, thus completing the cycle.

Very good electrolytic results are obtained through continuous operation, keeping the acid content at about 14 percent and the copper at 3½ percent. We are, however, at present operating intermittently, regenerating the acid content rather high (about 20 percent) and running the copper down to about 1 percent.

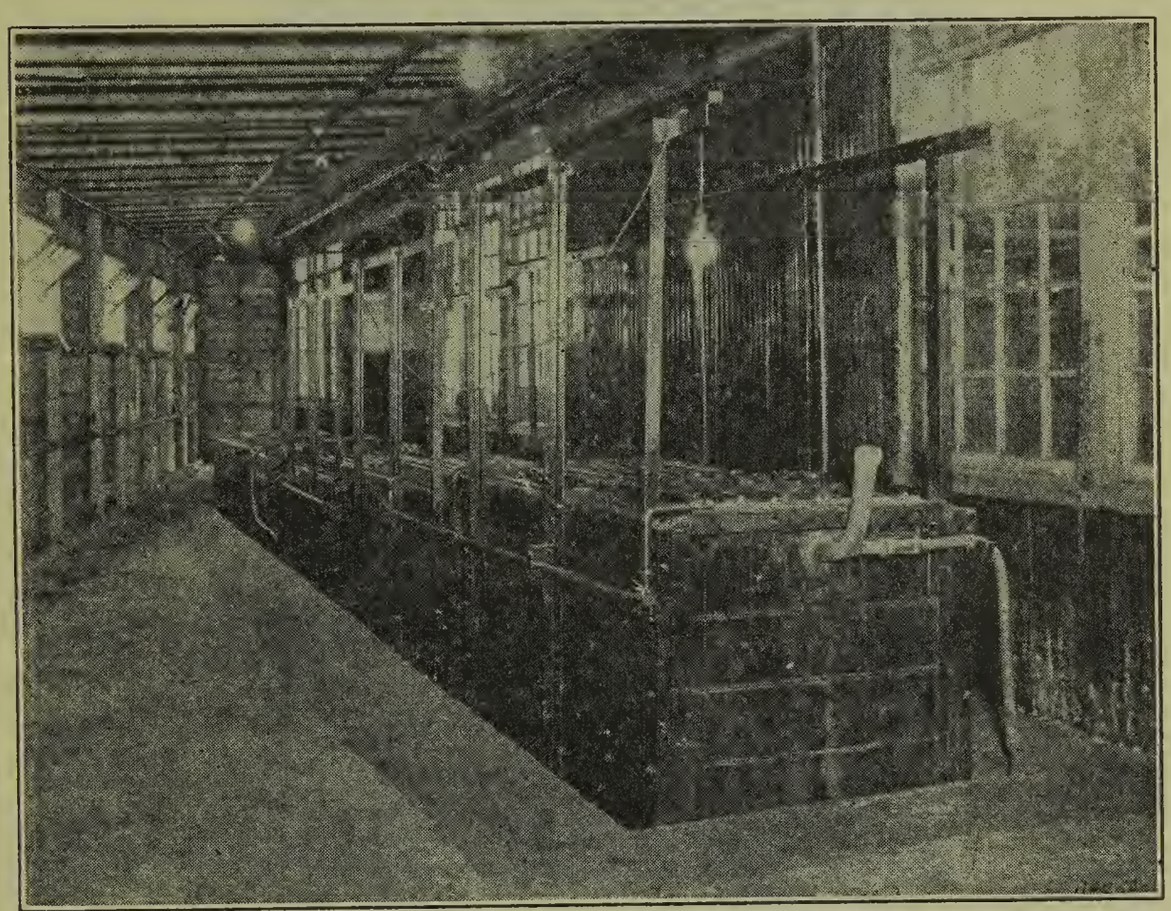


FIG. 3.

This plan is followed because of insufficient steam to satisfactorily operate the siphon which is relied upon to circulate the electrolyte. Only three tanks have been used for the electrolysis so far, the other two being held in reserve. At present one of these (see Fig. 4) is used for depositing a very heavy coating of copper on steel plates that are stamped up into discs for making arc lamp electrodes. These plates are first struck up in a cyanide bath and then the plating finished in this tank, using the waste pickling acid as the electrolyte and cathode plates from the recovery tanks

for the anodes. This application was suggested by Mr. C. W. Johnson, general superintendent.

A trolley is placed over the recovery tanks and serves to lift out the anodes and cathodes, which are placed in frames so that the entire set may be removed at one time. The anodes are of 7 lb. (350 g. per sq. dm.) sheet lead. They are 3 feet 10 inches (115 cm.) long by 2 feet 4 inches (70 cm.) wide and weigh 52 lb.

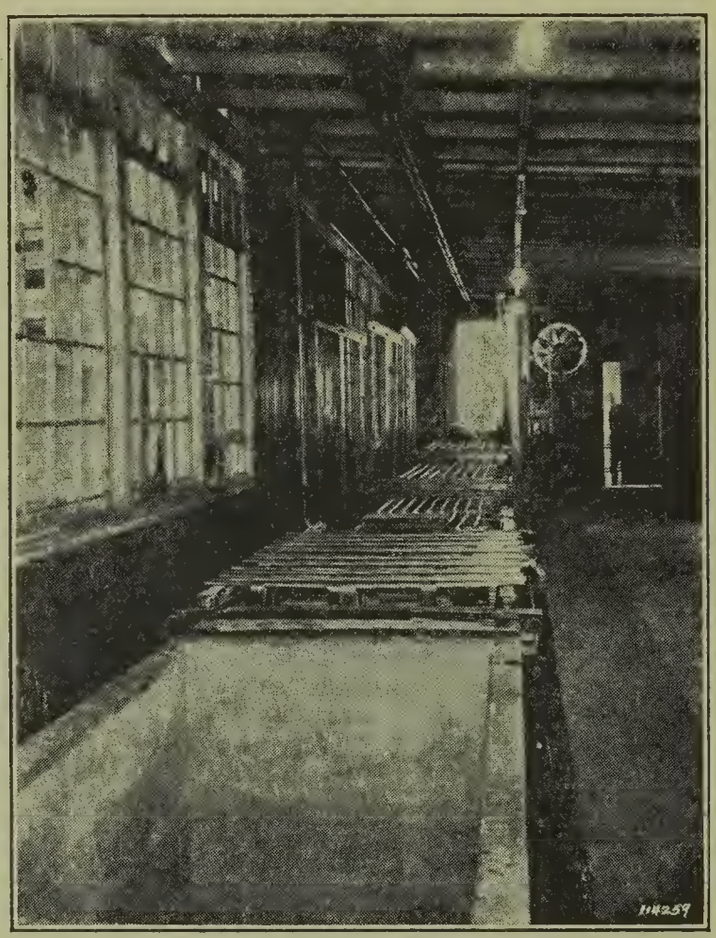


FIG 4.

(24 kg.) each. The starting sheets are at present of thin sheet copper, but will in the future be made by electrodeposition as is customary in electrolytic copper refineries.

The recovery tanks are arranged in cascade and set in a cement-lined pit provided with a sump to take care of any leakage. The series system is used, with parallel circulation across the face of the electrodes.

Rubber connectors, so made that they may easily be slipped off and replaced, connect the tanks with each other. They are cleaned

out from time to time, as there is some electrical leakage through them that plates out the copper in them and causes short circuits that burn out the rubber.

Current is supplied by a 60 volt—1,000 ampere generator, which has capacity for a considerably increased production. Fig. 5 gives the wiring diagram of the plant.

Our data on the preliminary operation of the plant is not very complete, but sufficient to show fairly satisfactory results. The plant has been run 60 days of 10 hours each, using 65 kilowatts, or a total of 1,600 kilowatt days. There was recovered 3,355 lb.

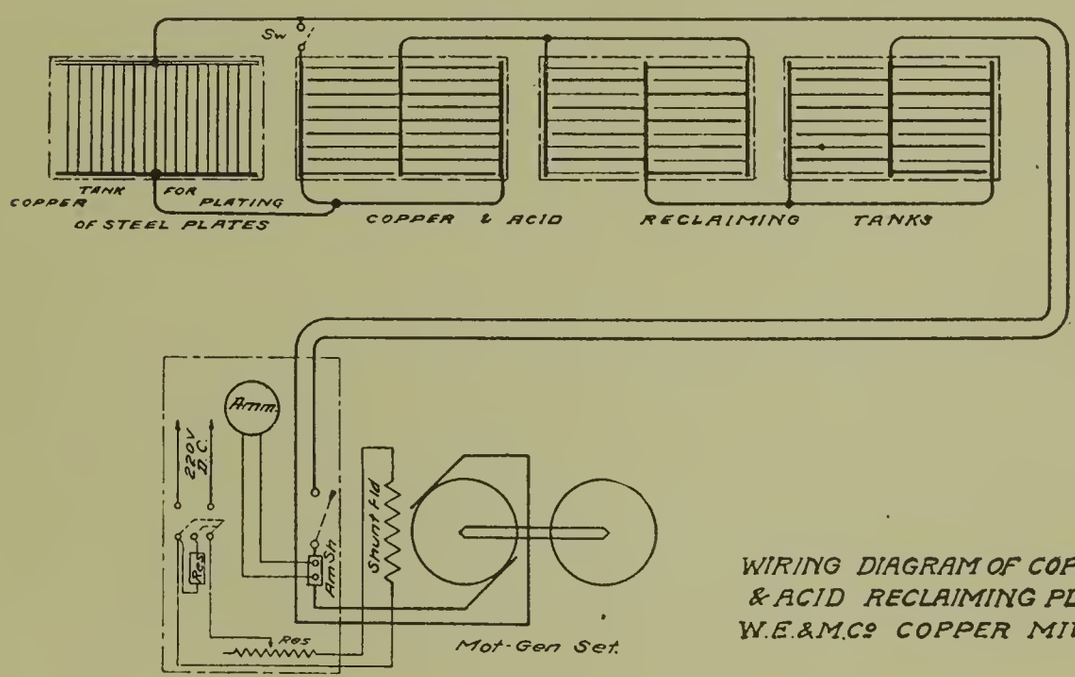


FIG. 5.

(1,525 kg.) of copper and in addition 420 lb. (190 kg.) of copper was plated upon 519 steel plates, making a total of 3,775 lb. (1,716 kg.) of copper recovered. This is slightly less than 1 lb. (453 g.) of copper per kilowatt hour.

The amount of acid recovered was about 5,500 lb. (2,500 kg.). The most marked advantage, however, was the increased rapidity of the pickling operation. The amount of copper pickled in the time mentioned was 2,714,594 lb. (1,233,900 kg.). Without the recovery plant it would have been necessary to considerably increase the capacity of the pickling tanks to do this amount of work.

Using three tanks in series and a current of 1,000 amperes at 2 volts, 135 lb. (61.4 kg.) of copper and 200 lb. (91 kg.) of acid

are recovered daily, showing an ampere efficiency of 72 percent. By adding additional tanks the output of the plant can be increased to 1,200 lb. (545 kg.) of copper and 1,800 lb. (818 kg.) of acid, as the generator has a capacity of 60 kilowatts.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

ELECTROLYTIC PICKLING PROCESS AND ITS EFFECT ON THE PHYSICAL PROPERTIES OF IRON AND STEEL

By John Coulson.*

[Abstract.]

To determine the embrittling effect of absorbed hydrogen upon steel pickled in different ways, samples of spring steel wire were pickled chemically by simple dipping in acid, also by electrolytic pickling, using them as cathodes or as anodes. Oscillation tests on the original wires, the same sand-blasted, and the three sets treated as described, showed the chemically cleaned and the cathodically cleaned to be very brittle, while the anodically cleaned had the strength of the original samples. Similar tests were also made with drill-rod steel, hot-rolled Bessemer, and cold-rolled steel, after which mechanical tests were made as to strength, elongation and reduction of area. The chemically and the cathodically cleaned specimens again showed greater brittleness, which was most marked with the high-carbon steels and less marked with the soft steel.

Brittleness of steel springs resulting from acid pickling and electroplating is a well-known phenomenon. Tempered springs which are pickled even for a few seconds in an acid bath snap when subjected to strain. Such springs, however, can be electrolytically pickled without destroying their resiliency. This paper cites an electrolytic process for removing oxides from the surface of iron, steel and other metals without impairing their physical properties.

It is known that metals absorb gases and that the amount absorbed depends upon the nature of the gas, the condition and character of the metal and the surrounding medium. These

* Physical Chemist, Westinghouse Research Laboratory, East Pittsburgh, Pa.

absorbed gases appear to play an important rôle in the behavior of iron, steel and certain alloys. Electrolytic iron is extremely brittle due principally to occluded hydrogen, and there appears no doubt that the brittleness of steel springs after pickling is due to the absorption of hydrogen during the ordinary pickling process. This effect is still more pronounced if the springs are made the cathode in an acidulated electrolyte.

Numerous experiments were carried out to determine the behavior of steel springs, drill rod, tempered steel, Bessemer and cold-rolled steel rods after undergoing different pickling treatments. The pickling bath consisted of a 27 percent solution of H_2SO_4 kept at 60° C. Test specimens of each of the above materials were treated consecutively; one-third of each set was pickled by the ordinary chemical process, one-third electrolytically as anode, and one-third as cathode. The lead tank containing the pickling bath served for the second electrode in the electrolytic processes.

The springs here dealt with were of 3/32 inch (2.4 mm.) diameter tempered steel wire wound closely in the form of a helix ¾ inch (19 mm.) diameter. Twenty of these springs constituted a set for the testing machine. The machine was adjusted to exert a maximum tension of about 16 lb. (7.3 kg.) on each spring, and then dropped to zero again. The number of oscillations of the machine and the life of each spring was automatically recorded.

Table I gives the history of one of the many sets of springs tested. Each set of springs was divided into groups of four, each group was constituted of springs of like treatment. The first group tabulated received no treatment further than tempering, which was common to all. The second group was sand-blasted; the third group was pickled 40 seconds chemically; the fourth group was pickled 40 seconds electrolytically as cathode, and the fifth group was pickled 40 seconds as anode. All the springs pickled chemically, and as cathode electrolytically, broke during the preliminary test, which consisted of a continuous strain for 12 hours. The crystalline structure of the breaks resembled that of glass-hard steel. No difference appeared to exist in the properties of the other groups of springs. All withstood, on the average, about ten million oscillations before breaking.

TABLE I.

Life Test on Steel Springs.

No.	Treatment	Load Lb.	Life (No. of Oscillations)
1	Original state	16.0	11,090,000
2		15.8	191,426
3		15.8	Not broken
4		15.8	13,173,000
5	Sand-blasted	16.1	10,017,000
6		15.6	13,715,000
7		15.8	8,373,210
8		16.0	11,495,000
9	Pickled 40 seconds chemically	16.0	Broke during preliminary test
10		15.4	
11		16.0	
12		15.6	
13	Pickled 40 seconds as cathode	15.8	45,000
14		15.8	Broke during preliminary test
15		15.9	
16		15.3	
17	Pickled 40 seconds as anode	16.0	14,220,000
18		16.0	1,714,000
19		15.8	12,387,000
20		15.8	11,725,000

Bending tests were carried out on bars 1 by ¼ inches (2.5 x 0.6 cm.) cold rolled steel. Test pieces which had been pickled chemically and electrolytically as cathode, showed fatigue and cracks much sooner than untreated test pieces or pieces which had been pickled electrolytically as anode. The records for the last two cases were very similar. The Brinell test for hardness indicated an increase of about 5 percent for specimens pickled as cathode, while no material change was noticeable for any of the others.

PHYSICAL TESTS.

The tensile strength, percent elongation and percent reduction, were determined for a number of rods of different materials, some of which had been pickled chemically, others electrolytically. Table II gives the results of a few of these tests. Each number tabulated is the average value for three rods similarly treated, and as a rule the values for a given set of rods all agreed fairly well.

TABLE II.

Material	Diam. mm.	Diam. inches	Pickling	Unit Stress Kg./sq. mm.	Unit Stress Lb./sq. in.	Reduction of Area percent	Elongation in 2 In. (5 cm.) percent
Spring steel wire	2.0	0.0785	None	178	254,000	51	..
	"	"	Chemical	173	247,000	3	..
	"	"	Cathode	173	247,000	3	..
	"	"	Anode	176	252,000	51	..
	5.2	0.205	None	160	228,000	47	..
	"	"	Cathode	157	224,000	3	2
	"	"	Anode	155	221,000	49	10
Drill rod....	6.3	0.25	None	118	169,000	22	5
	"	"	Cathode	113	162,000	7	2.5
	"	"	Anode	114	163,000	25	6.6
Hot rolled Bessemer..	5.9	0,236	Cathode	48	69,000	22	29
	"	"	Anode	47	68,000	63	35
Cold rolled steel	6.3	0.25	None	74	106,000	50	..
	"	"	Chemical	70	100,000	40	..
	"	"	Cathode	72	103,000	35	..
	"	"	Anode	71	102,000	50	..

It will be seen from the table that all the materials pickled either chemically or electrolytically as cathode show deleterious effects. The properties of the high carbon steel undergo the greatest modification. The majority of specimens of this material tested showed no elongation or reduction whatever when pickled electrolytically as cathode. Anode pickling, it appears, does not impair the physical properties of any of the materials here dealt with.

Fig. 1 illustrates typical breaks of the spring steel 0.205 inch (5.2 mm.) in diameter, before and after pickling. Rod *A* was tested in its original state, *B* after pickling electrolytically as anode and *C* as cathode. The fracture of *A* and *B* are perfectly cup shaped; the test records of these specimens also agreed closely. *C*, on the other hand, shows a very short break without any appreciable reduction in area.

GAS PENETRATION.

Fig. 2 shows the rupture of ¼-inch (6mm.) cold-rolled steel before and after pickling. These test pieces were cut from the

same rod; *E* and *F* were pickled, each 40 seconds, electrolytically as anode and cathode respectively, while *D* was tested in its original condition. It will be seen from the figure that *D* and *E* have cup breaks essentially alike; further, their test records also agreed. *F*, however, shows an irregular break with a dark center encircled by a bright band. This band is well defined, and indicates clearly the depth the hydrogen penetrated the metal during the process of pickling. The rate at which the hydrogen penetrates the metal

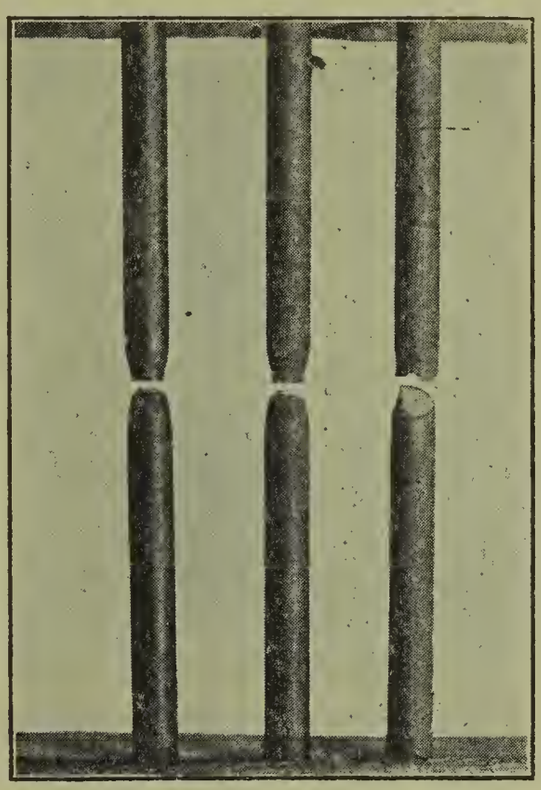


FIG. 1.

increases with the current density. In the case of ordinary chemical pickling absorption takes place more slowly.

It is very probable that the hydrogen absorbed by the metal, where it loses its identity as nascent hydrogen, forms an alloy, thus modifying the physical properties of the steel. The bright band of the test piece *F*, Fig. 2, indicates that the rupture took place along the crystal faces and not through the crystals. The light is here reflected from the crystal faces and is, therefore, more intense than the reflection from the ruptured crystals of the central area.

Test specimens, made brittle by pickling either chemically or

electrolytically as cathode, were restored to the original condition by anneàling. This restoration was effected considerably by re-pickling the test pieces electrolytically as anode. A number of test pieces of spring steel 0.205 inch (5.2 mm.) diameter were pickled two minutes electrolytically as cathode; half of these broke with no elongation and no reduction. The other half was re-pickled two minutes electrolytically as anode, and then tested. These had an average value for elongation and reduction of 5.5 percent and 25 percent respectively. Non-pickled material showed

D E F

FIG. 2.

8 percent elongation and 40 percent reduction. It is probable that prolonged re-pickling electrolytically as anode would restore completely the physical properties of the metal.

SUMMARY.

Hydrogen is absorbed rapidly by iron and steel when pickled either chemically or electrolytically as cathode, in acid solutions; thus causing brittleness, diminution of the tensile strength, elongation and reduction of area of the material.

Iron and steel may be cleaned effectively and quickly by pickling electrolytically as anode, without impairing their strength, resiliency or other physical properties.

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Westinghouse Research Laboratory,
East Pittsburgh, Pa.
August, 1917.

DISCUSSION.

CARL HERING[1]: This paper does injustice to cathode pickling. There is no doubt that it would make highly-tempered hardened steel, brittle; I should not suppose anybody would use it for that purpose. The large field for cathode pickling is for tonnage steel, because the scale is thereby dissolved and the steel itself is protected against being dissolved, while with anodic pickling the iron under the scale is dissolved at the cost of good metal and acid. In the ordinary chemical pickling plants the scale collects in large quantities in the pickling vats, showing that it is not dissolved, but as a cathode it goes into solution freely.

It is therefore not fair to condemn cathodic pickling in general, simply because it is not applicable to hard, tempered steel. Moreover, anodic pickling which the author seems to recommend would surely not be applicable in many cases. It is incorrect in principle, in that the scale, of course, is not dissolved, it being already an oxide; the only possible cleansing action in this case would be dissolving the good metal beneath the scale. In anodic pickling the oxidizing action would tend to form an oxide on the surfaces

[1] Consulting Electrical Engineer, Philadelphia, Pa.

to be cleaned, which I should think would interfere greatly with subsequent tinning or galvanizing.

As far as the development of hydrogen in the iron is concerned, cathodic pickling is on a par with the ordinary chemical pickling; in the latter the hardening, if there is any, in the case of tonnage steel like sheets and wires, seems to be of no importance, and in the annealing, this occluded hydrogen, if there is any, leaves the metal again. In subsequent tinning or hot galvanizing, the escape of this hydrogen during that operation may even be very advantageous.

W. RUDER[2]: Near the end of the paper the author refers to the nature of the rupture of the crystals, and says, "The bright band of the test piece indicates that the rupture took place along the crystal faces and not through the crystal. The light is here reflected from the crystal faces, and is therefore more intense than the reflection from the ruptured crystals of the central area." Has this been accurately determined? The brightness would not determine that. It would have to be examined more closely to determine whether the surface were plane or conchoidal. The importance of this lies in its throwing light on the nature of the compound formed by the action of the hydrogen on the steel. If the fracture is conchoidal, that is, is granular, that action of the hydrogen would be on the intercrystalline material and not on the iron itself.

As a matter of fact, we have found that even molecular hydrogen has the effect of producing intergranular brittleness in steel. This kind of fracture, if treatment is made at high temperatures (1,300° C.), is unnatural for steel. However, the nature of the fracture should throw some light on the effect produced.

PRESIDENT C. G. FINK[3]: As I understand Mr. Ruder, he wants to point out that the embrittling effect is on the intergranular material rather than on the crystal itself.

W. RUDER: I have no direct data one way or the other, except the effect of molecular H at high temperatures.

J. W. RICHARDS[4]: We are again in danger of making too broad a generalization. The title of the paper should be changed to

[2] General Electric Co., Schenectady.
[3] Director of Research, Chile Exploration Co., New York City.
[4] Professor of Metallurgy, Lehigh University.

"Physical Properties of Carbon Steel"; it would then refer to the kind of metal tested. The conclusions at the end of the paper should also state that the results apply to carbon steel. Wherever the electrolyte penetrates into the pores of the metal, the surfaces wetted are the seat of the electrochemical action. Therefore, the absorption of hydrogen is much more active where the solution has penetrated under the outer surface and has come in contact with the interior surfaces. The more the carbon content the greater is this effect.

JOHN COULSON (*Communicated*): Dr. Carl Hering remarked that injustice had been done to cathode pickling in my paper. My purpose was not to discredit any particular pickling process, but to show the effects of the foregoing processes on the physical characteristics of iron. His statement: "That the scale is not dissolved, it being already an oxide; the only possible cleansing action in this case would be dissolving the good metal beneath the scale," referring to anode pickling, is incorrect. From an elementary consideration it will be seen that while pickling as anode the scale is partially dissolved due to the action of the acid on the iron, liberating hydrogen which unites with part of the oxygen formed electrolytically. The remainder of the oxygen is mostly given off as gas, a very small quantity used in attacking good metal. There is no iron oxide left on the metal, hence no harmful effect in subsequent tinning or galvanizing. A very small amount of the original scale is thrown to the bottom of the electrolyte. The facts brought out in the paper are that hardening and extreme brittleness result from cathode pickling.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

THE PREVENTION OF BRITTLENESS IN ELECTROPLATED STEEL SPRINGS

By T. S. Fuller.

[Abstract.]

The literature of the relations of hydrogen to iron, causing brittleness, is extensively reviewed. In the author's experimental work he tested the breaking strength of seven untreated springs, then tested similar ones electrically copper plated, pickled in sulphuric acid, and tinned by dipping in molten tin and then copper plated. The pickling makes them brittle, also copper plating, but tin-dipping before copper plating is found to prevent embrittlement entirely.

One of the serious problems which confronts the makers and users of non-corroding steel springs is the brittleness produced therein during electroplating. The embrittlement is generally conceded to be caused by the nascent or atomic hydrogen which is liberated at the cathode, although M. DeKay Thompson and C. W. Richardson[1] conclude from their experiments that hydrogen is not the cause of this trouble.

Roberts-Austin[2] gives a brief summary of what is known concerning the relations of hydrogen and iron, and describes an investigation carried on by him. The cooling curve of the hydrogen-iron alloys shows the existence of two distinct evolutions of heat, which occur at about 487° and 261° C. (908° and 502° F.) After repeated heatings of the metal in vacuo, these points become so small that it is impossible to identify them with certainty. It is remarkable that repeated heatings and coolings of low-carbon and

[1] Met. and Chem. Eng., 16, 83-84 (1917).

[2] Fifth Report to the Alloys Research Committee: Steel, 1899.

electrodeposited iron in vacuo reduce the magnitude of all the now well-known molecular changes—Ar 1, Ar 2, Ar 3, as indicated on the cooling curves. The exhausted electro-iron may be recharged with electrolytic hydrogen, by making it the negative electrode of a cell decomposing acidulated water. If the recharged iron be heated in vacuo, as already described, it will be found that the evolutions of heat in cooling, and consequently the points on the curves, will be restored to their original magnitude.

Heyn[3] found that iron heated above 730° C. in hydrogen and then quenched in water or in mercury became brittle. Iron similarly heated in nitrogen and quenched was not brittle.

Wedding[4] discusses in detail the relations of iron and hydrogen and develops the theory that the two elements form unstable alloys. The fact that iron takes up nascent hydrogen at low temperatures and is made very brittle thereby, was developed by a number of investigators about 1870-80. Numerous researches showed that the flexibility and elasticity of iron wire and steel springs were greatly decreased by pickling, and the effect is the more marked, the thinner the material. Electrolytic iron may contain nearly 250 times its volume of hydrogen, while the commercial iron averages only 0.5 its volume.

Burgess and Engle[5] found that unheated electrolytic iron dissolved in normal sulphuric and hydrochloric acid almost twice as rapidly by weight as commercial zinc. After heating to 1,000° C. and cooling slowly, the rate of solution became as low as the least corroded form of iron tested, *i. e.*, transformer iron.

Richards and Behr[6] found that nascent hydrogen is more readily absorbed by iron than is gas. They mention earlier observations of the same thing: "Cailletet and Johnson showed in 1875 that iron will take up at ordinary temperatures electrolytic hydrogen deposited upon it, while ordinary gaseous hydrogen has no effect. Johnson's work was particularly interesting. His iron was in the form of soft wire and his simple method of testing for hydrogen was to bend the wire to test its brittleness, since he had discovered

[3] Stahl und Eisen, **20**, 837 (1900).
Z. Phys. Chem., **58**, 760-1 (1907).
[4] Internationale Kongress angew. Chem., **2**, 27-51 (1903).
[5] Trans. Amer. Electrochem. Soc., **9**, 199-206 (1906).
[6] Carnegie Institution of Washington Publication No. 61, Part II (1906).

the interesting fact that hydrogen imparted this property to iron previously tough. Immersing the wire in acids which react on it to form hydrogen uniformly made it brittle. Making the wire the cathode in neutral, acid, or alkaline solutions had the same effect, but iron anodes were unaffected even in the acid solution. Other wires were placed in a bottle full of water, and hydrogen was made to bubble violently through the water, without any trace of absorption." The occluded hydrogen is slowly given off, upon exposure to the air, a little more rapidly in pure water and quickly in ferrous sulphate. The active hydrogen raises the single potential of iron by as much as 0.15 volt, although hydrogen gas has a lower potential than iron.

Burgess[7] says: "Among the properties of electrolytic iron which may give it some added usefulness is its content of hydrogen and the brittleness which results from this occluded gas. This hydrogen may be of some service in reducing oxide in a melt. The brittleness of the electrolytic iron before the hydrogen is driven off makes it easy to break it into pieces suitable for introduction into the steel crucibles."

Charpy and Bonnerot[8] have observed that iron at ordinary temperatures acts as a membrane, permeable to nascent but not to molecular hydrogen. This was observed by experiment with hydrogen liberated from attack of acid on the iron membrane, and also by making the latter cathode solution. Osmosis occurs only when the hydrogen is liberated directly upon the membrane. Pressures as high as 14 atmospheres have been produced by such osmosis into a closed chamber.

Andrew[9] concludes from experiments upon iron and caustic soda that hydrogen, upon being occluded by iron, immediately gives rise to the production of large crystals and brittleness. "It is not contended that hydrogen *per se* is the cause of the crystallization, but rather that it is due to the evolution or occlusion of hydrogen effecting a molecular disturbance."

"Iron gives up the greater part of its hydrogen as the temperature passes through that of the critical stage."

[7] Chem. Engr., **13**, 82 (1911).
Trans. Amer. Electrochem. Soc., **19**, 181-3 (1911).
[8] Comp. Rend., **154**, 592-4 (1912); **156**, 394-6 (1913).
[9] Trans. Faraday Soc., **9**, 316-29 (1914).

He quotes Stromeyer[10] to the effect that contact with cold caustic soda of a concentration of over 90 or 100° Twaddell not only causes mild steel to become brittle, but also tubes and plates crack and rivet heads fly off. This takes place even out of contact with the air where there is little corrosion. Stromeyer states that this brittleness and corrosion occur only when the metal is in tension. Unstressed or compressed metal is not affected.

Guillet[11] mentions that electrolytic iron as deposited is extremely brittle and hard, "due to the occluded hydrogen, and probably also, as will be seen, to interstrain."

Storey[12] found that the extremely brittle and soluble electrolytic iron, heated to between 500 and 600° C. gave off a large amount of hydrogen and became ductile and more resistant to acid without undergoing any apparent change in crystalline structure.

Baraduc-Muller[13] conducted preliminary experiments on the gases contained in liquid steel. Ingots were melted and the gases pumped off and analyzed. In one case the gas amounted to 54.2 percent by volume of the metal. Hydrogen was the principal constituent. The mechanical properties were improved by removal of the gas.

Parr[14] has studied the embrittling action of sodium hydroxide on soft steel, and he concludes "The evidence seems to show that the embrittling effect of sodium hydroxide on steel is due to the evolution of hydrogen and the absorption by the steel of the hydrogen in the nascent state."

EXPERIMENTAL WORK.

The work herein described was done to find, if possible, a means of preventing the brittleness of electroplated steel springs. All of the test samples were made from the same lot of black spring wire 0.120 inch (30 mm.) in diameter. Eight or ten turns of the wire were wound on a ⅛ inch (30 mm.) mandrel and 3 inch (7.5 cm.) lengths left on both ends of the spring to fit into the grips of the testing machine. This necessitated a sharp bend at

[10] J. Iron and Steel Inst., 1, 1909.
[11] J. Iron and Steel Inst., **90**, 66-80 (1914).
[12] Trans. Amer. Electrochem. Soc., **25**, 489-504 (1914).
[13] Rev. Metal, **12**, 804-8 (1915).
[14] University of Illinois, Engineering Experiment Station, Bulletin No. 94 (1917).

both ends of the coil and it was at one of these bends where the break usually occurred. The breaking strength as determined by an Olsen testing machine proved to be the best criterion for comparing the springs treated in different ways.

Effect of Copper-Plating.

The breaking strength of seven untreated test springs (1–7, Table I) was determined. Five of the seven samples broke above 400 lb. (182 kg.), the values ranging, however, between 310 and 439 lb. (141 and 200 kg.).

Two like springs were made cathode for ½ hour in a cyanide copper solution. These (No. 8 and No. 9, Table I) broke at 69 and 137 lb. (31 and 62 kg.), the treatment in the copper-plating solution having cut down the breaking strength to 17 and 35 percent, respectively, of the average value of the seven untreated springs. Furthermore, the copper-plated samples stretched but very little before breaking, while the untreated ones stretched to two or three times their original length.

Effect of H_2SO_4 Pickle.

Test spring No. 10 (Table I) was pickled for three minutes and No. 11 (Table I) for one hour, in a twenty percent H_2SO_4 solution. The former sample broke at 198 lb. (90 kg.), showing a reduction in strength to 50 percent of the untreated value, and the latter at 29 lb. (13 kg.) with a reduction in strength to 7 percent of the untreated value. The latter sample was reduced 0.001 inch (0.025 mm.) in diameter by solution in the acid.

Effect of Tin in Preventing Brittleness.

It was then thought that a coating of tin on the steel might prove to be impervious to the action of the atomic hydrogen, and thus allow the spring to be copper-plated without impairing its mechanical properties. This was found to be true. The first test samples which were tin-dipped, however, were pickled for a few seconds in H_2SO_4 previous to the dipping. These (12 and 13, Table I) showed breaking strengths of only 38 and 32 lb. (17 and 14.5 kg.). Nos. 14 and 15, which were pickled, tin dipped, and

copper-plated, showed breaking strengths of 22 and 34 lb. (10 and 15.5 kg.). The very poor results obtained from these four samples, however, were due to the H_2SO_4 pickle which they received before dipping.

Two samples (16 and 17, Table I), without having been pickled, were dipped directly into rosin and then into molten tin heated to a temperature of 260°–300° C. These springs broke at 300 and 352 lb. (136 and 160 kg.), or in the neighborhood of the range of the untreated springs (310–439 lb. (141–200 kg.).

Samples 18, 19, 20, 21, 22 and 23 (Table I) were sand-blasted, dipped in rosin and molten tin, and copper-plated in the same solution used for the copper-plated springs previously described. These samples showed breaking strength from 318 to 417 lb. (145 to 189 kg.), all of which are well within the range of the values for the untreated springs.

TABLE I.

		Breaking Strength			
1.	Untreated	433	lb.	(196	kg.)
2.	"	438	"	(199	")
3.	"	435	"	(197	")
4.	"	374	"	(170	")
5.	"	439	"	(200	")
6.	"	418	"	(190	")
7.	"	310	"	(141	")
8.	Copper plated	69	"	(31	")
9.	" "	137	"	(62	")
10.	Pickled 20 percent H_2SO_4—3 minutes	198	"	(90	")
11.	" " " " —1 hour	29	"	(14	")
12.	" Sn dipped	38	"	(17	")
13.	" " "	32	"	(15	")
14.	" " " Cu plated	22	"	(10	")
15.	" " " " "	34	"	(16	")
16.	Sn dipped (rosin) Cu plated	300	"	(136	")
17.	" " " " "	352	"	(160	")
18.	Sand blasted, Sn dipped (rosin) Cu plated	390	"	(178	")
19.	" " " " " " "	318	"	(145	")
20.	" " " " " " "	417	"	(190	")
21.	" " " " " " "	356	"	(162	")
22.	" " " " " " "	410	"	(186	")
23.	" " " " " " "	402	"	(184	")

TABLE II.

No. 24.—Untreated.

Diameter of wire (inch)..... 0.117
Mean diameter of spring..... 0.773
Number of turns..... 5

Load, Pounds	Stretch, Inches	Total Set, Inches
0	0	0
20	0.078	0
40	0.250	0
60	0.344	0
80	0.516	0
100	0.703	0.062
120	1.062	0.234
140	1.625	0.687

No. 25.—Copper Plated.

Diameter of wire (inch)..... 0.120
Mean diameter of spring..... 0.782
Number of turns..... 5

0	0	0
20	0.125	0
40	0.312	0
60	0.453	0
80	0.578	0
100	0.703	0.078
120	1.031	0.234
140	1.656	0.734

No. 26.—Tin Coated (Rosin) Copper Plated.

Diameter of wire (inch)..... 0.123
Mean diameter of spring..... 0.779
Number of turns..... 5

0	0	0
20	0.156	0
40	0.281	0
60	0.406	0
80	0.531	0.0156
100	0.656	0.0625
120	0.984	0.2187
140	1.406	0.5312

Effect of Tin Dipping on the Mechanical Properties.

The mechanical properties of the samples were not appreciably changed by the tin treatment. Table II shows the results of tests made on three similar, but differently treated springs, made from

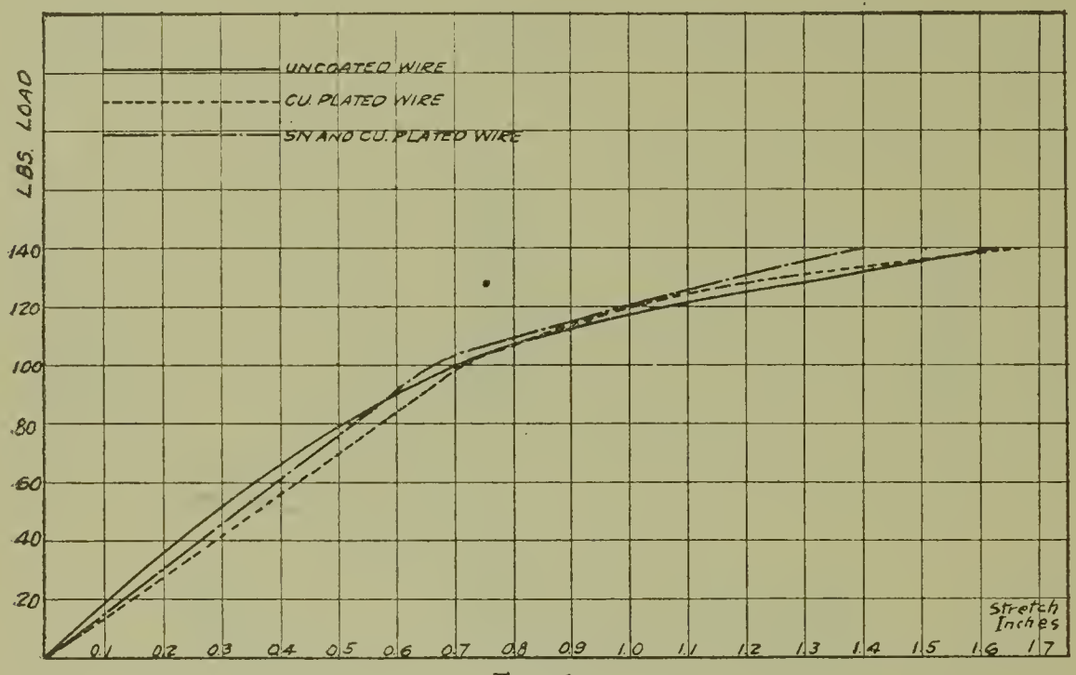

FIG. 1.

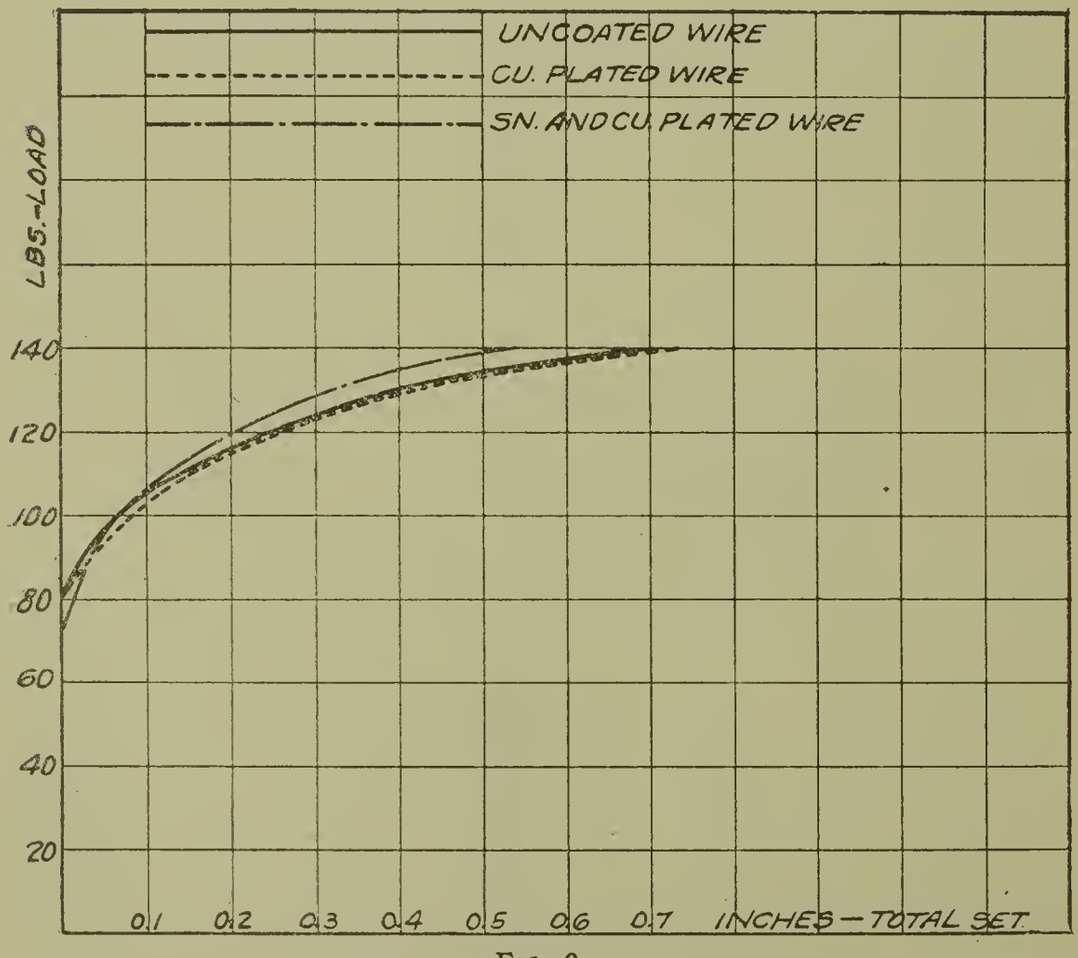

FIG. 2.

the same lot of wire as those previously described, but wound on a ½ inch (12.5 mm.) instead of a ⅛ inch (3 mm.) mandrel. No. 24 was untreated, 25 copper-plated and 26 tin-dipped (rosin) and copper-plated. For the same load, all three springs show approximately the same stretch and total set.

The three curves in Fig. 1 show graphically the relation of the stretch to the load for springs 24, 25 and 26, plotted from data in Table II.

Fig. 2 shows the relation of the permanent set to the load for the same springs.

CONCLUSIONS.

The facts are all in accord with the assumption that the absorption by the steel of atomic or nascent hydrogen liberated at the cathode is the cause of the embrittlement of steel springs in the plating bath.

This embrittlement may be prevented by first dipping the springs in a bath of molten tin. The tin forms a coating on the steel which is impermeable to atomic hydrogen. Rosin may be used as a flux in the tin dipping. The springs may be sand-blasted, but must not be pickled in acid.

Steel springs may be dipped in molten tin at a temperature of 260°–300° C. without appreciably changing their mechanical properties.

Research Laboratory,
General Electric Co.,
Schenectady, N. Y.

DISCUSSION.

G. B. HOGABOOM[1]: There is a tendency to accept any formula for a plating solution that may be in use at the time, electroplating steel springs in it, and not taking into consideration the effect of such solution on steel. Papers have been published in the Transactions of this Society upon nickel-plating solutions in which hydrogen has been deposited primarily. In such a solu-

[1] Research Laboratory, Scovill Mfg. Co., Waterbury, Conn.

tion the steel springs will become brittle. By correcting the solution so that the hydrogen would not be deposited primarily, the brittleness of the spring would not occur.

PRESIDENT C. G. FINK[2]: Have you, Mr. Fuller, tried to plate your springs and avoid the formation of such a deposit?

MR. FULLER: I have not tried that. I have used only the ordinary cyanide solution.

MR. HOGABOOM: Has Mr. Fuller analyzed the solution to determine the amount of free cyanide present?

T. S. FULLER (*Communicated*): Mr. Hogaboom's remarks refer to a phase of the subject quite different from that described in my paper. If the average electro-plater be given a lot of steel springs to plate, he will in most cases produce a brittle product, and one which cannot be relied upon. I have described a preliminary way of treating steel springs, after which they may be plated by any electro-plater and found dependable. I have not, however, gone into the question of the composition of the plating solution.

[2] Director of Research, Chile Exploration Co., New York City.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

THE CORROSION OF METALS BY ACIDS.

By Oliver P. Watts and Newton D. Whipple.

[Abstract.]

The authors study the rate of solution of zinc, amalgamated zinc, iron, copper, silver, and gold, in various solutions, at normal atmospheric pressures and under a vacuum, and draw many theoretical deductions therefrom regarding the discharge potential of hydrogen on various metals and the electrochemical mechanism of the corrosion.

On account of the great economic interests involved, the corrosion of metals is one of the most important of the many technical problems which today engage the attention of electrochemists. The importance of galvanic couples in the corrosion and protection of metals received early recognition, but it is only in recent years that the whole problem of corrosion has been recognized as being electrochemical in its nature. It was formerly supposed that zinc dissolved in sulphuric acid because the affinity of this metal for the SO_4 radical exceeded that of hydrogen for the same; and that for the action to go on until either the zinc or the acid was exhausted it was only necessary that the product, zinc sulphate, should be soluble.

Among the many natural phenomena to whose explanation the theory of electrolytic dissociation has been applied, is the corrosion of iron[1]; according to this, the formation of a soluble compound is not a prerequisite to the dissolving of a metal by an acid, the metal going directly into solution as ions, and any compounds which may be formed being the result of combinations which occur after solution has taken place. From either the chemical

[1] W. R. Whitney, J. Amer. Chem. S. (1903), 1, 394.

or the ionic point of view, an electrolyte is like a room so crowded that no newcomers can enter unless some of those already present pass out to make place for them.

On account of the enormous scale on which it occurs, the rusting of iron has received more attention than any other case of the corrosion of metals, and it is admitted by all that oxygen is necessary for the continuation of this process. Since the passage of ions into solution does not demand oxygen, it appears at first sight as if the proved need for oxygen were fatal to the ionic theory of the corrosion of metals.

In the extraction of gold and silver by the cyanide process, the presence of oxygen was also found to be necessary in order that solution of the metals may take place with sufficient rapidity to make the process practical. In this case the products formed by the dissolving of the metals do not contain oxygen, and from either the chemical or the ionic view of the dissolving of metals, the function of oxygen was obscure. The extensive use of the cyanide process led to a detailed study of the chemical changes involved, and in 1903 the rôle of oxygen in the dissolving of gold by cyanide solutions was made clear for the first time by that pioneer mining engineer (who perished in the Titanic disaster) Henry F. Julian,[2] who showed by experiment that the accelerative effect of oxygen on the solution of gold by cyanide is due to its removing from the surface of the ore the hydrogen which is displaced by the gold when it dissolves. If the hydrogen is allowed to accumulate its presence greatly retards solution of the gold.

Several years later, W. H. Walker[3] gave a similar explanation of the function of oxygen in the corrosion of iron. "We have shown that the primary function of oxygen in the corrosion of iron is in depolarizing those cathodic portions of the iron upon which hydrogen tends to precipitate, and that a secondary function is the oxidation of the ferrous iron ion to the ferric form, with its subsequent precipitation as ferric hydroxide."

When iron is put in electrical contact with metals of higher potential, such as zinc or cadmium, the iron is cathode in the voltaic cell that is formed whenever the combination of metals is

[2] Cyaniding Gold and Silver Ores. Julian and Smart, p. 65.
[3] J. Amer. Chem. S. (1907), 1264.

moistened, and therefore the iron corrodes less rapidly than if it were not in contact with the other metal. But when iron makes contact with copper, silver, or platinum, metals of lower potential, the iron is anode in the voltaic cell, and its corrosion is greater than it would be in the absence of the other metals. Landis[4] says, "Do not plate with a nobler metal a material that is likely to be subjected to wear on one spot, and which spot is to be moistened with liquids." But in order that galvanic action shall take place it is usually unnecessary for electroplated objects to be subjected to sufficient use to wear off the plating at some point, since electro-deposits of ordinary thickness are sufficiently porous to permit galvanic action as soon as the object is exposed to moisture.[5]

The excessive corrosion of iron by sulphuric acid caused by adding salts of copper, silver, or platinum, is just what would be predicted from the theory of galvanic action. Cases are known, however, in which the addition to sulphuric acid of salts of other metals, also more noble than iron, instead of stimulating corrosion of the iron, greatly retards it.[6] Compounds of arsenic have this effect, and have been used with good results in commercial work to prevent corrosion of iron pipes by acid waters.[7] The reason for this unusual protective action is the high "discharge potential"* of hydrogen on arsenic. The action is as follows: Since iron is electro-positive to arsenic, it displaces this element from solution, and a porous coating of arsenic is formed on the iron. In the voltaic cell thus formed arsenic is the cathode, and consequently it is on this that the hydrogen expelled by the dissolving of iron is deposited. The discharge potential of hydrogen on

[4] Trans. Am. Electrochemical Soc. (1911), **19**, 62.

[5] Trans. Am. Electrochemical Soc. (1916), **30**, 145.

[6] Trans. Am. Electrochemical Soc. (1912), **21**, 337.

[7] Corrosion and Preservation of Iron and Steel. Cushman and Gardner, p. 297.

* The term "discharge potential" has been used so little in recent electrochemical literature that an explanation of this useful term may be desirable at this time. If copper be used as cathode in a solution of ferrous sulphate until completely covered with iron, a change in potential takes place from —0.51, the potential of copper, to +0.09, that of iron. If sulphuric acid is used as the electrolyte instead of copper sulphate, hydrogen is deposited in place of copper, and the potential rises only to —0.01 volt, when hydrogen is visibly evolved; the potential of the cathode when this occurs is the discharge potential of hydrogen on copper. The potential of the cathode required for the evolution of hydrogen depends on the material of which it is made, so that the discharge potential of hydrogen is different for each metal. It is earnestly recommended that "discharge potential" be used instead of overvoltage in cases where the latter term has heretofore been employed to designate the potential of an electrode required for the evolution of some particular gas.

arsenic exceeds the potential of iron in dilute sulphuric or hydrochloric acid, and hence as iron dissolves, thereby causing an accumulation of hydrogen on the arsenic, the deposition of this gas becomes more difficult, and before enough hydrogen has been displaced to escape as visible bubbles, the potential required for its further deposition equals the driving force, the potential of iron, and action therefore ceases.

Various explanations have been given for the protection afforded by amalgamation. Grove in 1837[8] suggested that the protection is due to polarization, the hydrogen that is evolved combining with the mercury to render it electro-positive. Faraday considered that the mercury produced a uniform condition on the surface, and thus afforded protection. Carhart[9] considers the protective effect to be two-fold: 1st, the elimination of local currents by the production of a film of pure zinc on the surface, and 2d, the adhesion of a film of hydrogen to the amalgamated surface.

In the references already given, the function of oxygen in the dissolving of gold and iron have been explained and the importance of hydrogen in this process has been brought out. It is only reasonable to expect that the principles which have been found to apply to the solution of gold and iron will hold for the dissolving of other metals, and it was with the hope of extending the application of these principles to the corrosion of metals generally that the experiments which follow were undertaken.

Unless otherwise specified, the tests were carried on in tumblers containing 180 or 190 c.c. of the acid or other corroding agent. The tumblers were covered by watch glasses to lessen evaporation, and were set in a constant temperature water bath. The square sheets of metal, with the exception of gold and platinum, were of such dimensions that they were held upright by the sides of the tumblers, so that both sides of the specimen were exposed to the corroding agent.

One of the authors[10] has already presented to this Society an account of experiments which show that the protection of iron by arsenic against corrosion by sulphuric acid is probably due to

[8] Phil. Mag., **15**, 81.
[9] Primary Batteries, p. 34.
[10] Trans. Am. Electrochemical Soc. (1912), **21**, 337.

the discharge potential of hydrogen on arsenic being higher than the potential of iron in this acid. It has been stated[11] that the protective action of arsenic on iron is much less in hydrochloric than in sulphuric acid.

If, as is believed by the authors, the protective action of arsenic is due to the polarizing action of a film of hydrogen on the arsenic, the addition to the acid of oxidizing agents capable of removing hydrogen should wholly or partially, according to the amount of oxidizing agent used, nullify the protective effect of arsenic.

TABLE I.

Temperature of bath.....37.5° C.
Time .....45 hours
Area of specimens.....60 sq. cm.

Specimen	Reagents used	Loss in Grams
1. Fe	N. HCl 190 c.c.	4.9812
2. Fe	N. HCl 190 c.c., 5 c.c. 30% H_2O_2	5.3355
3. Fe	N. HCl 190 c.c., 0.25 g. Na_3AsO_4	0.2631
4. Fe	N. HCl 190 c.c., 0.25 g. Na_3AsO_4, 5 c.c. H_2O_2	2.9706
5. Fe	N. H_2SO_4 190 c.c.,	4.5245
6. Fe	N. H_2SO_4 190 c.c., 5 c.c. 30% H_2O_2	5.2119
7. Fe	N. H_2SO_4 190 c.c., 0.25 g. Na_3AsO_4	0.1050
8. Fe	N. H_2SO_4 190 c.c., 0.25 g. Na_3AsO_4, 5 c.c. H_2O_2	2.8118

From the experiments of Table I it is seen that corrosion in hydrochloric acid was slightly greater than in sulphuric acid, agreeing with the reaction velocities of these acids in other cases. The oxidizing agent induced extensive corrosion in No. 4 and 8, in spite of the presence of arsenic.

If the authors' hypothesis is correct, in solutions which corrode iron without evolution of hydrogen, arsenic should have no protective action. The results of corrosion by ferric chloride and ferric sulphate are given in Tables II and III.

TABLE II.

Temperature of bath.....37.5° C.
Time .....45 hours
Area of specimens.....58 sq. cm.

Specimen	Reagents used	Loss in Grams
9. Fe	N. HCl 190 c.c.	3.0315
10. Fe	N. HCl 190 c.c., 15 g. $Fe_2(SO_4)_3$	5.8159
11. Fe	N. HCl 190 c.c., 15 g. $Fe_2(SO_4)_3$, 0.25 g. Na_3AsO_4	1.6088
12. Fe	H_2O 190 c.c., 15 g. $Fe_2(SO_4)_3$	1.9326
13. Fe	H_2O 190 c.c., 15 g. $Fe_2(SO_4)_3$, 0.25 g. Na_3AsO_4	1.6276

[11] Trans. Am. Electrochemical Soc. (1905), 8, 169.

Table III.

Temperature of bath.....38° C.
Time .....46 hours
Area of specimens.....58 sq. cm.

Specimen	Reagents used	Loss in Grams
14. Fe	N. HCl 190 c.c., 10 g. $FeCl_3$	5.4359
15. Fe	N. HCl 190 c.c., 10 g. $FeCl_3$, 0.25 g. Na_3AsO_4	1.7528
16. Fe	H_2O 190 c.c., 15 g. $FeCl_3$	1.6552
17. Fe	H_2O 190 c.c., 15 g. $FeCl_3$, 0.25 g. Na_3AsO_4	1.4576

Table IV.

Specimen	Reagents used	Loss in Grams
18. Fe	H_2O 190 c.c., 10 g. $FeCl_3$	0.9501
19. Fe	H_2O 190 c.c., 10 g. $FeCl_3$, 0.25 g. Na_3AsO_4	1.0058
20. Fe	H_2O 190 c.c., 10 g. $FeCl_3$, 0.25 g. Na_3AsO_4, 5 c.c. H_2O_2	1.1608

A comparison of 12 with 13, and 16 with 17, shows such a slight protection against corrosion by ferric salts as might be expected from the lessened surface of iron exposed, due to some iron being covered by arsenic, but 18-19 shows a contrary effect; this discrepancy can not at present be explained. The corrosion produced by the combination of hydrochloric acid and ferric sulphate considerably exceeds the sum of the losses in those reagents separately. This is in agreement with results previously obtained[12] in which iron lost 2.84 grams in sulphuric acid, 4.22 grams in copper sulphate, and 14.89 grams in a combination of the two solutions. Table II shows the arsenic to have a selective action; it stops corrosion by acid, but does not interfere with corrosion by ferric sulphate, except to the small extent previously noted as probably due to a reduction in the surface of iron exposed. Nos. 19 and 20 show that the great acceleration in the corrosion of iron protected by arsenic, which was observed when hydrogen peroxide was added to acids, does not occur in ferric chloride; no hydrogen liberated in the process of corrosion, no acceleration produced by the oxidizing agent.

For more than a half-century battery zincs which are to be used in acid electrolytes have been amalgamated, to prevent useless loss of zinc when no current is being drawn from the cell. Several explanations of the protection afforded by amalgamation have already been quoted, from which it appears that hydrogen is in some way generally held responsible for the protective effect. A

[12] Trans. Am. Electrochem. Soc. (1912), **21**, 350.

comparison of the discharge potentials of hydrogen on zinc and on mercury with the potential of zinc makes clear the nature of protection by amalgamation. The discharge potential of hydrogen on zinc as computed from Caspari's values for overvoltage in sulphuric acid[13] is 0.462, slightly lower than the potential of zinc, so that pure zinc should dissolve slowly in sulphuric acid; but the discharge potential of hydrogen on mercury is 0.542, slightly higher than the potential of zinc.

When amalgamated zinc is put into dilute sulphuric acid, zinc begins to dissolve, and displaces hydrogen just as if the metal were unamalgamated; but before the deposited hydrogen has become sufficient in amount to be visible or to escape as gas, the polarization produced by it, and therefore the electrical pressure necessary to deposit more hydrogen, has risen to equal the potential of zinc, when this metal is no longer capable of displacing hydrogen, and its solution ceases. If this view is correct, addition to the acid of an oxidizing agent that is capable of removing hydrogen ought to cause amalgamated zinc to corrode, as should also the use of a corroding agent that evolves no hydrogen by its action. The results of such tests on commercial sheet zinc are given in Table V.

TABLE V.

Temperature of bath.....37 5° C.
Time .....45 hours
Area of specimens.....60 sq. cm.

Specimen	Reagents used	Loss in Grams
21. Zn	N. HCl 190 c.c.	6.3462
22. Zn	N. CH_3COOH 190 c.c.	1.3928
23. ZnHg	N. HCl 190 c.c.	0.6718
24. ZnHg	N. HCl 190 c.c., 10 g. $K_2Cr_2O_7$	3.0527
25. ZnHg	N. HCl 190 c.c., 10 g. $NaClO_3$	1.9926
26. Zn	N. HCl 190 c.c., 2 g. $HgCl_2$	—0.3038
27. Zn	N. HCl 190 c.c.	6.1117
28. Zn	N. HCl 190 c.c., 2 g. $HgCl_2$, 5 c.c. H_2O_2	2.2844
29. Zn	H_2O 190 c.c., 10 g. $FeCl_3$	1.3895
30. ZnHg	H_2O 190 c.c., 10 g. $FeCl_3$	1.5840

It is seen from Table V that oxidizing agents stimulated the corrosion of amalgamated zinc, just as was expected, and that amalgamation does not protect zinc against corrosion by ferric chloride. No significance should be attached to the fact that the

[13] Zeit. f. phys. Chem., 30, 89.

corrosion of amalgamated zinc by hydrochloric acid was about ten percent of that dissolved from the unamalgamated metal, for in the latter case the acid was all used up, so that had double the amount of acid been employed, the corrosion would have been only five percent of that of the unprotected zinc. In 26 there was a gain in weight, due to the replacement of some of the zinc by mercury.

As a further confirmation of the failure of amalgamation to protect zinc from corrosion by reagents which do not liberate hydrogen, the experiments of Table VI were tried, using ammonium and potassium persulphates as corroding agents. The results came as a surprise after so many experiments had shown that neither arsenic nor mercury protects from corrosion unless hydrogen is evolved to cause polarization. Samples 31, 32 and 33 show that corrosion in ammonium persulphate is cut in half by amalgamation, and that the addition of hydrogen peroxide stimulates corrosion. Nos. 34, 35 and 36 show a similar result except that protection by amalgamation was much greater in potassium persulphate. There was an evolution of gas in every case.

TABLE VI.

Temperature of bath.....37.5° C.
Time .....107 or 45 hours
Area of specimens.....60 sq. cm.

Specimen	Reagents used	Loss in Grams
31. Zn*	H_2O 190 c.c., 10 g. $(NH_4)_2(SO_4)_2$.....	2.5160
32. ZnHg*	H_2O 190 c.c., 10 g. $(NH_4)_2(SO_4)_2$.....	1.0186
33. ZnHg*	H_2O 190 c.c., 10 g. $(NH_4)_2(SO_4)_2$, 5 c.c. H_2O_2....	2.8530
34. Zn	H_2O 190 c.c., 10 g. $K_2(SO_4)_2$.....	2.2894
35. ZnHg	H_2O 190 c.c., 10 g. $K_2(SO_4)_2$.....	0.1589
36. ZnHg	H_2O 190 c.c., 10 g. $K_2(SO_4)_2$, 5 c.c. H_2O_2.....	2.1700

*Time 107 hours

In the experiments with ferric chloride and ferric sulphate no gas had been evolved; it was therefore thought that the discordant results obtained in the experiments of Table VI were in some way due to the gas evolved, which was naturally supposed to be oxygen. The gas set free in the reaction between zinc and ammonium persulphate was collected, and found to be hydrogen, free from oxygen, so that the protection by amalgamation and the stimulation of corrosion by hydrogen peroxide is in harmony with

previous experiments. The large loss of weight of amalgamated zinc in ammonium persulphate indicates either a less evolution of hydrogen or a much more active oxidation of this than occurred in the potassium persulphate.

As a result of these experiments it is believed that the protection of zinc by amalgamation is due to the elevation of the discharge potential of hydrogen brought about by the presence of mercury. In the absence of oxidizing agents any metal will be protected by amalgamation when its potential in the solution used exceeds the discharge potential of hydrogen on itself, but is less than the discharge potential of hydrogen on mercury.

Whether viewed from the standpoint of Nernst's theory of solution pressure or from a consideration of their potentials, the metals copper, mercury, silver, platinum and gold fall into a different class as regards corrosion by acids (except for such as are oxidizing agents) than do the metals so far considered. Their solution tensions are less, and their potentials are lower than the values published, and usually accepted for hydrogen. This means that none of these metals should be able to displace hydrogen ions from electrolytes, and hence that they should not be attacked by acids.

The violent attack of copper and vigorous solution of silver by strong nitric acid seemed to the authors to mean that the displacement of hydrogen from acids by these metals must begin, just as with zinc or iron; but before the hydrogen has reached a sufficient concentration to be evolved as visible gas the potential required for its further discharge exceeds that of the metal, so that action ceases unless this incipient film of hydrogen is removed. When its removal is brought about by any means, enough metal immediately dissolves to cause the replacement of the films by expulsion of hydrogen from the acid. If the film of hydrogen be continuously and rapidly removed the solution of metal will take place at the same rate. No other view seems capable of reconciling the vigorous attack of these metals by nitric acid, and their comparative immunity to attack by sulphuric acid. If this theory is correct it ought to be possible to dissolve silver and copper rapidly by adding an oxidizing agent to any strong acid that forms a soluble salt with these metals.

In Table VII are given the potentials of several metals according to measurements by Neumann[14], the discharge potential of hydrogen calculated from values of overvoltage by several experimenters, and the heats of formation of chlorides.[15]

TABLE VII.

Potentials in normal			Disch. Potential of Hydrogen		Potential minus Dis. Potential of Hydrogen		Heats of formation of chlorides		
	SO_4	Cl						Anhydr.	In water
Zn	0.524	0.503	Hg	0.548	Fe	0.183	K_2,Cl_2	211220	202340
Cd	0.162	0.174	Zn	0.468	Zn	0.062	Zn,Cl_2	97210	112840
Fe	0.093	0.087	Pb	0.408	Ni	0.006	Cd,Cl_2	93240	96250
Sn	....	—0.085	Sn	0.298	Cd	—0.080	Fe,Cl_2	82050	99950
Pb	....	—0.095	Cd	0.248	Sn	—0.377	Sn,Cl_2	80790	81140
H	—0.238	—0.249	As	0.158	Pb	—0.497	H_2,Cl_2	.....	78630
Cu	—0.515	....	Cu	—0.002	Cu	—0.507	Pb,Cl_2	82770	75970
As	....	—0.550	Ni	—0.022	As	—0.702	Cu,Cl_2	51630	63710
Hg	—0.980	....	Ag	—0.082	Ag	—0.759	Hg,Cl_2	54490	51190
Ag	—0.974	....	Pt	—0.112	Pt	—1.022	Ag_2,Cl_2	58760	.....
Pt	....	—1.140	Au	—0.212	Au	—1.138	H_2,Cl_2	44000	.....
Au	....	—1.356	PtPt	—0.232	Hg	—1.522	$\frac{2}{3}(As,Cl_3)$	40927	48840
							$\frac{2}{3}(Au,Cl_3)$	15213	18113

The displacement of one metal from solution by another can be predicted from the values of their potentials; a metal precipitates from solution all metals of lower potential than itself. Neumann has succeeded in precipitating on a sheet of platinum by hydrogen all metals whose potentials are below hydrogen in this table. It might therefore be expected that all metals above hydrogen would displace this gas, and therefore dissolve readily in all acids whose salts of the metal are soluble, and that the metals below hydrogen would not be attacked by acids. But the value —0.238, wrongly called the potential of hydrogen, is really the discharge potential of hydrogen on platinized platinum; the change which has been brought about in the potential of platinum by causing hydrogen to be deposited on it measures the polarization of platinum by hydrogen. Since the amount of polarization by hydrogen varies with different metals, in order to determine from theoretical considerations whether or not a metal should displace hydrogen from acids it is necessary to compare the potential of the metal with the discharge potential of hydrogen on that particular metal.

[14] Z. f. phys. Chem. (1894), 14, 203.
[15] Thermo-Chemistry, Thomsen, tr. by K. A. Burke (1908).

If the potential of a metal exceeds the discharge potential of hydrogen on it, this metal should dissolve readily in acids whose salts of the metal are soluble, but if its potential is less than the discharge potential of hydrogen, acids should dissolve it only at the rate at which the displaced hydrogen dissolves in the acid, is removed by oxidizing agents, or is otherwise disposed of. To test the correctness of these generalizations several metals of the latter class, *viz.*, lead, tin, copper, silver, gold and platinum, were treated with acids, with and without the addition of oxidizing agents.

TABLE VIII.

Temperature of bath.....37.5° C.
Time .....45 hours
Area of specimens.....60 sq. cm.

Specimen	Reagents used	Loss in Grams
37. Pb	N. CH_3COOH 190 c.c.	0.2143
38. Pb	N. CH_3COOH 190 c.c., 0.25 g. Na_3ASO_4	0.0490
39. Pb	N. CH_3COOH 190 c.c., 2 g. $HgCl_2$	0.8529
40. Pb	N. CH_3COOH 190 c.c., 2 g. $HgCl_2$, 5 c.c. H_2O_2	6.5596
41. Pb	N. CH_3COOH 190 c.c.	0.2019
42. Pb	N. CH_3COOH 190 c.c., 5 c.c. H_2O_2	6.4026
43. PbHg	N. CH_3COOH 190 c.c.	0.1515
44. PbHg	N. CH_3COOH 190 c.c., 5 c.c. 30% H_2O_2	5.5071

Table VIII shows a slow rate of solution of lead in acetic acid, which is a measure of the rate of removal of hydrogen from the surface of the metal—probably by the air dissolved in the acid. The effect of amalgamation in diminishing corrosion is trifling, as would be expected, since it only raises slightly the discharge potential of hydrogen, which already exceeded the potential of lead. The oxidizing agent had its usual effect and enormously increased corrosion, this time in every case in which it was used, since pure lead, unlike zinc and iron, can corrode only so fast as the hydrogen is removed from it.

Similar experiments with copper and tin (Table IX) show these metals to be even less attacked by sulphuric acid than was lead by acetic in the last experiments; allowing for the shorter time, attack by hydrochloric acid is about the same as the dissolving of lead by acetic acid. These metals are readily dissolved in the presence of oxidizing agents, except for 54, in which a fine, white precipitate indicated the formation of an insoluble compound. It has been shown that when iron or copper is hammered or cold-

worked the potential is raised, and the tendency to corrode is increased. A comparison of 47 with 49 indicates that this is probably true of tin also.

TABLE IX.

Temperature of bath.....38° C.
Time .....19 or 22 hours
Area of specimens.....64 or 60 sq. cm.

Specimen	Reagents used	Loss in Grams
45. Cu	N. HCl 180 c.c.	0.1171
46. Cu	N. HCl 180 c.c., 10 g. $KMnO_4$	4.6084
47. Sn† cast	N. HCl 180 c.c.	0.0947
48. Sn† cast	N. HCl 180 c.c., 10 g. $KMnO_4$	3.6160
49. Sn† hammered	N. HCl 180 c.c.	0.1155
50. Cu*	N. H_2SO_4 180 c.c.	0.0226
51. Cu*	N. H_2SO_4 180 c.c., 10 g. $K_2Cr_2O_7$	2.8637
52. Cu*	N. H_2SO_4 180 c.c., 10 g. $NaClO_3$	4.3084
53. Sn*†	N. H_2SO_4 180 c.c.	0.0373
54. Sn*†	N. H_2SO_4 180 c.c., 10 g. $K_2Cr_2O_7$	0.0171
55. Sn*†	N. H_2SO_4 180 c.c., 10 g. $NaClO_3$	8.3576

* Time 19 hours.
† Area of specimens 64 sq. cm.

The noble metals, silver, gold and platinum, are similar to copper and tin in that their potentials are less than the discharge potential of hydrogen upon them. These metals should therefore be but slightly attacked by acids unless an oxidizing agent is present. Tests with these metals are given in Tables X and XI.

TABLE X.

Temperature of bath.....37.5° C.
Time .....45 hours
Area of specimens.....60 sq. cm.

Specimen	Reagents used	Loss in Grams
56. Ag	N. H_2SO_4 190 c.c.	0.0015
57. Ag	N. H_2SO_4 190 c.c., 5 c.c. 30% H_2O_2	1.6953
58. Ag	N. CH_3COOH 190 c.c.	0.0016
59. Ag	N. CH_3COOH 190 c.c., 5 c.c. 30% H_2O_2	1.7997

Although some silver dissolved in the pure acids, the rate of corrosion is less than for any metal previously tested; the stimulating effect of the hydrogen peroxide was also less than heretofore. These facts indicate that much less hydrogen is displaced by silver before equilibrium is reached than in case of the metals previously tested.

TABLE XI.

Temperature of bath.....37.5° C.
Time .....45 hours
Area of specimens.....4, 12 or 24 sq. cm.

Specimen	Reagents used	Loss in Grams
60. Au	N. H_2SO_4 190 c.c.	0.0017
61. Au	N. H_2SO_4 190 c.c., 5 c.c. H_2O_2	0.0018
62. Au	N. H_2SO_4 190 c.c., 5 g. $NaClO_3$	0.0004
63. Au	N. H_2SO_4 190 c.c., 5 g. $KMnO_4$	0.0000?
64. Au*	N. HCl 190 c.c.	0.0008
65. Au*	N. HCl 190 c.c., 5 c.c. H_2O_2	0.0070
66. Au*	N. CH_3COOH 190 c.c.	0.0005
67. Au*	N. CH_3COOH 190 c.c., 5 c.c. H_2O_2	0.0005
68. Pt†	N. HCl 190 c.c.	0.0000
69. Pt†	N. HCl 190 c.c., 5 c.c. H_2O_2	0.0006
70. Pt†	N. HCl 190 c.c., 5 g. KNO_3	0.0002

* Area of specimens 12 sq. cm.
† Area of specimens 4 sq. cm.

The gold in 63 had a purplish coating, which because of the thinness of the metal, could not be removed before weighing, hence it is probable that there was a trace of gold dissolved in this case. With the exception of 65, which is a special case and will be referred to later, there was no appreciable solution of gold or platinum, and oxidizing agents were without their usual stimulating effect on corrosion. Thinking that the velocity of reaction might be sufficiently stimulated by more concentrated acid and increase of temperature to make corrosion visible, the experiments of Table XII were conducted with gold leaf in an evaporating dish which was heated over a Bunsen burner.

The addition of a chloride or a bromide to a mixture of oxidizing agent and acid caused prompt and complete solution of the gold. Aside from 73, in which there is a possibility of the liberation of chlorine by decomposition of the acid, there was only one case, 72, in which the gold dissolved without the presence of a chloride, bromide or iodide, and in this the action was so slow that many hours were required to dissolve the bit of gold leaf. No tests were made for traces of dissolved gold, since the purpose of these experiments was to determine if in the presence of oxidizing agents other acids would dissolve gold at a rate comparable to that at which it is attacked by aqua regia, and at which all other metals used in these tests (except platinum) have been corroded when oxidizing agents were present.

Table XII.

The Solution of Gold in Acid Plus Oxidizing Agents.

	Acid	Temp.	Time	Oxidizer	Result
71.	H_2SO_4 30%	90	15 m.	CrO_3	Gold leaf remains
	H_2SO_4 30%	70	10 m.	$KMnO_4$	Gold leaf remains
	H_2SO_4 30%	90	....	HNO_3	Gold leaf remains
	H_2SO_4 30%	..	....	$NaNO_2$	Gold leaf remains
	H_2SO_4 30%	..	....	KBr	Gold dissolved
72.	H_2SO_4 80%	150	20 m.	CrO_3	Gold remains
	H_2SO_4 80%	Cold	24 h.	CrO_3	Gold dissolved
73.	$HClO_4$	80	....	HNO_3	Gold remains
	$HClO_4$	95	....	$NaNO_2$	Gold remains
	$HClO_4$	95	....	CrO_3	Gold dissolved
74.	HBF_4	Hot	....	HNO_3	Gold remains
	HBF_4	Hot	....	KBr	Gold dissolved
75.	H_3PO_4	98	5 m.	CrO_3	Gold remains
	H_3PO_4	98	....	$NaNO_3$	Gold remains
	H_3PO_4	..	....	HNO_3	Gold remains
	H_3PO_4	..	....	KI	Gold slowly dissolved
	H_3PO_4	..	....	KBr	Gold quickly dissolved
76.	H_3PO_4	90	....	$KMnO_4$	Gold remains
77.	H_3PO_4 + H_2SO_4 50%	115	....	CrO_3	Gold remains
	H_3PO_4 + H_2SO_4 50%	142	....	CrO_3	Gold remains
78.	H_3PO_4	130	30 m.	CrO_3	Gold remains
	H_3PO_4	Boiling	80 m.	CrO_3	Gold remains
	H_3PO_4	..	....	NaCl	Gold dissolved
79.	HCl 15%	60	....	CrO_3	Gold quickly dissolved

The dissolving of gold by other reagents than aqua regia has been recorded by many experimenters. Comey[16] states that gold is dissolved by concentrated sulphuric acid to which either potassium permanganate or iodic acid has been added; and that selenic acid is the only single acid that dissolves the metal. Lenher[17] found that gold dissolves in sufficient amount to respond to chemical tests in either hot concentrated sulphuric or phosphoric acid containing potassium permanganate, lead peroxide, manganese dioxide, or nitric acid, and that in some of these solutions gold dissolves even at zero degrees centigrade.

The great difference in the rate of solution of gold by aqua regia or any combination of a halogen acid with an oxidizing agent, in comparison with its rate of dissolving in other acids and

[16] Dictionary of Solubilities, p. 172.
[17] J. Amer. Chem. S. (1904), 26, I, 550.

an oxidizing agent, seems to indicate some fundamental difference in the nature of the dissolving of gold by aqua regia and by the other acids. The effect of oxidizing agents in stimulating the dissolving of lead, tin, copper and silver in acids indicates that these metals displace an appreciable amount of hydrogen, the rate of removal of which by the oxidizing agent fixes the speed of dissolving of the metal. Granting the solution of gold by sulphuric or phosphoric acid and oxidizing agents, the very slow rate of action indicated by these experiments in comparison with the action of similar solutions on the metals just mentioned means a correspondingly slow oxidation of the displaced hydrogen. Since the oxidizing agents were as powerful, and in several cases the same as used with the other metals, the slow rate of oxidation of hydrogen must be due either to the presence at any instant of only an extremely small quantity of hydrogen on the gold, or to the replacement of the oxidized hydrogen at this very slow rate. There is no reason for thinking that gold can displace hydrogen from hydrochloric acid more rapidly or to a much greater extent than from other strong acids; therefore the rapid solution of gold by aqua regia shows that this is not a case of the displacement of hydrogen by the metal, but, like the dissolving of iron by ferric chloride, a reaction in which hydrogen is not evolved. The starting point of the reaction must be the oxidation of the hydrogen of the acid; after which solution of the gold may be conceived to occur either chemically, by combination with nascent chlorine, or ionically, by electrostatic attraction between chlorine and gold ions.

Gold and platinum, being readily dissolved only by aqua regia or a solution of a halogen acid and an oxidizing agent, are thus distinguished from lead, copper, tin, and silver, with which they were at first classed by the authors.

A study of Table VII shows an interesting relation between heats of formation and the results of these experiments. In aqueous solution the heat of formation of hydrochloric acid exceeds that of the chlorides of copper, lead, mercury, gold, and of course of silver and platinum, and hence none of these metals should be capable of displacing hydrogen from water solution of hydrochloric acid; but in the anhydrous condition the heat of formation of hydrochloric acid is less than that of all of the above chlorides, except those of arsenic, gold and platinum, so that, when

water is left out of consideration, all of these metals except the three last mentioned should displace hydrogen from hydrochloric acid. The facts are in accordance with this. When water is allowed to play its part, copper, lead, mercury, silver, gold and platinum are not readily dissolved by non-oxidizing acid solutions; but when the hydrogen of water is taken care of by an oxidizing agent all of the above, except gold and platinum, dissolve in acids, provided their salts are not insoluble. A classification of the metals according to whether or not their potentials exceed the discharge potential of hydrogen on them leads to the same grouping as does a consideration of the heats of formation in aqueous solution; and experiments with acids containing oxidizing agents draw the dividing line between the two classes at the same point as does a consideration of the heats of formation of the anhydrous chlorides, *viz.*, just below silver.

The third set of data in Table VII, obtained by subtracting the discharge potential of hydrogen from the potential of the metal, furnishes a rough index of the rate at which pure metals may be expected to dissolve in acids; when this index is negative the rate of solution is limited to the speed with which the displaced hydrogen is removed from the surface of the metal, and for rapid solution an oxidizing agent is necessary.

The necessity for supplying oxygen in the treatment of gold ores by cyanide has already been referred to. S. B. Christy[18] found the potential of gold to have the following values: in N.KCN + 0.37, in N/10 KCN + 0.23, in N/100 KCN + 0.09, and in N/1000 KCN —0.38 volt. The need for oxygen in dissolving gold signifies that the potential of gold in the solutions used in cyaniding gold ores is less than the unknown discharge potential of hydrogen on gold. To verify this, measurements were made in tenth normal potassium cyanide. No gas could be observed on immersing gold in this solution, but at a cathode potential of +0.42 volt (N.calomel electrode = —0.56) bubbles of hydrogen clung to the gold, and with the aid of a lens the escape of a tiny bubble was occasionally observed. On raising the impressed E. M. F. until the current density of 0.05 ampere per square decimeter was obtained, the potential of the cathode had increased to +0.57 volt, and the escape of gas was readily seen.

[18] Amer. Chem. J. (1902), **27**, 354.

The potential of gold in potassium cyanide is less than the discharge potential of hydrogen upon it; hence the necessity for oxygen in order to dissolve this metal in cyanide solutions.

The nature of protection by amalgamation has already been discussed in the light of these experiments. In a paper on "The Function of Oxygen in the Corrosion of Metals"[19] the prevention of the continued corrosion of iron by the film of hydrogen which is supposed to deposit on it is attributed to the high electrical resistance of hydrogen gas, and in discussing the accelerative effect of certain voltaic couples and of impurities on the rusting of iron, the same author attributes this to a catalytic oxidation of hydrogen by the cathode material or impurity. Speaking of the slight corrosion of zinc relative to that of iron in salt water, he says,[20] "The explanation consistent with the electrolytic theory is that the zinc does not dissolve and corrode, not because there was formed an adherent and protective layer of zinc hydroxide, but because the zinc does not catalyse the reaction $H_2 + O = H_2O$ with sufficient rapidity to continually remove the hydrogen from the surface of the zinc. Although the catalysing action of the iron surface is not so rapid as is the platinum surface, it has sufficient rapidity to depolarise the iron strip and to render continuous the solution, and therefore the corrosion of the iron. * * * The author has already shown that 'mill scale' or magnetic oxide of iron is strongly electro-negative to iron. Since mill scale is insoluble in water and cannot of itself enter into the reaction, its only function must be analogous to that of platinum or other insoluble conductor of this kind, *viz.*, to furnish a surface on which the hydrogen liberated by the dissolving of iron can separate and be catalytically oxidized to water again."

It is the opinion of the authors that the observed protection of iron by hydrogen is due, not to the *resistance* of the gas, but to the rise in potential produced by its presence on the iron. The protection of iron and zinc against corrosion, by contact with arsenic and zinc respectively, has been shown to be due to the high discharge potential of hydrogen on these metals; and the excessive corrosion of iron and zinc induced by contact with mill scale, silver, platinum, etc., is believed by the writers to be due

[19] W. H. Walker, Trans. Am. Electrochemical Society (1908), **14**, 178.
[20] Jour. Iron & Steel Inst. (1909), I, 75.

to a low value for the discharge potential of hydrogen on these materials, and not to any particular power of catalysing the oxidation of hydrogen. The real office of platinum as a stimulant to the corrosion of another metal is to furnish a cathode that is polarized to only a slight degree by the collecting of hydrogen on it, and which, together with the metal that is corroding, constitutes a cell of high working E. M. F.

The superiority of nitric acid over other mineral acids as the general solvent for metals has long been recognized. These experiments show that the special value of nitric acid, aside from nitrates being more generally soluble than other salts, lies in its being at the same time an acid and an oxidizing agent.

The foregoing experiments indicate that the inherent nature of the reaction between acids and metals is always the same. On immersion in an acid, metal begins to go into solution and hydrogen is displaced. But the law of mass action applies here. As hydrogen accumulates, the driving force required to deposit more of this gas increases, and if the potential, solution pressure, or whatever other term may be chosen to designate the relative displacing power of metals in electrolytes, be very low, like that of copper or silver, equilibrium is quickly reached and the action comes to a standstill. If the potential of the metal is high, as for zinc and iron, the reaction goes on, the density of the hydrogen film increases to such a point that gas begins to escape, and as equilibrium has not yet been reached, the reaction continues, as is usual in chemical changes when one of the products escapes from the field of action. The effect of oxidizing agents is to remove one product, hydrogen, and so prevent attainment of the equilibrium which is otherwise quickly reached with the metals of low potential, copper, mercury, silver, etc. If the oxidizing agent is a vigorous one it may remove hydrogen more rapidly than it normally escapes from the more positive metals, zinc and iron, and the rate of solution of these metals is accelerated. If hydrogen is removed from the metal very slowly, attack by the acid must also be slow, like the dissolving of silver in sulphuric and acetic acids. If some means be provided for removing hydrogen from the surface of the metal more rapidly than usual, the speed of dissolving is thereby increased. In every case the rate of solution

of metal by an acid is governed by the rapidity with which hydrogen is removed from the field of chemical action.

To supplement this study of the corrosion of metals by acids a few experiments were tried with alkalies. Because most of the metallic hydroxides are insoluble, only a few metals are available, from which zinc, tin and lead were chosen as representative.

Since hydrogen is the material displaced by the metal which dissolves, it was expected that the effect of amalgamation and of oxidizing agents on the rate of corrosion would be the same as in acids, except as modified by the changed potentials of the metals in this new electrolyte.

TABLE XIII.

Temperature of bath.....37.5° C.
Time .....45 hours
Area of specimens.....60 sq. cm.

Specimen	Reagents used	Loss in Grams
80. Sn	N. NaOH 190 c.c.	0.0302
81. Sn	N. NaOH 190 c.c., 0.25 g. Na_3AsO_4	0.0292
82. Sn	N. NaOH 190 c.c., 10 g. $KMnO_4$	0.6771
83. Sn	N. NaOH 190 c.c., 10 g. KNO_3	0.0300
84. Pb	N. NaOH 190 c.c.	0.1101
85. Pb	N. NaOH 190 c.c., 0.25 g. $NaAsO_4$	0.1026
86. PbHg	N. NaOH 190 c.c.	0.0850
87. Pb	N. NaOH 190 c.c., 5 g. $KMnO_4$	0.0978
88. Pb	N. NaOH 190 c.c., 5 g. KNO_3	0.0938
89. Pb	N. NaOH 190 c.c., 5 g. $NaClO_3$	0.0942
90. Zn	N. NaOH 190 c.c.	0.0363
91. Zn	N. NaOH 190 c.c., 0.25 g. $NaAsO_4$	0.1016
92. ZnHg	N. NaOH 190 c.c.	0.0326
93. Zn	N. NaOH 190 c.c., 5 g. $KMnO_4$	0.3318
94. Zn	N. NaOH 190 c.c., 5 g. KNO_3	0.7052
95. Zn	N. NaOH 190 c.c., 5 g. $NaClO_3$	0.0856

The first experiments, Table XIII, gave some unexpected results. The accelerative effect of oxidizing agents was much less than in acids, and varied greatly; not only did corrosion vary with different oxidizing agents, but also for the same agent with different metals. Potassium permanganate caused the greatest corrosion of tin, but with zinc this reagent proved less effective than potassium nitrate, which did not corrode tin at all. None of the oxidizing agents increased the corrosion of lead. Amalgamation decreased the corrosion of lead and zinc very slightly.

Arsenic stimulates the corrosion of zinc (90, 91) because the discharge potential of hydrogen on arsenic (see Table VII) is

much below that on zinc; it is a case of substituting a cathode which polarizes to a much less degree by hydrogen. Arsenic fails to stimulate the corrosion of lead and tin similarly (80, 81, 84, 85), because the potential of these metals in sodium hydrate is less than the discharge potential of hydrogen on arsenic; the voltaic cell formed by either of these metals with arsenic is completely polarized by hydrogen, and its E. M. F. becomes zero.

TABLE XIV.

Temperature of bath.....46° C.
Time .....45 hours
Area of specimens.....60 sq. cm.

Specimen	Reagents used	Loss in Grams
96. Sn	180 c.c. NaOH (200 g/L), 5 g. picric acid	3.7324
97. SnHg	180 c.c. NaOH (200 g/L)	0.0055
98. SnHg	180 c.c. NaOH (200 g/L), 5 g. picric acid	3.1014
99. Zn	180 c.c. NaOH (200 g/L)	0.0168
100. ZnHg	180 c.c. NaOH (200 g/L)	0.0130
101. Zn	180 c.c. NaOH (200 g/L), 5 g. picric acid	5.8750
102. ZnHg	180 c.c. NaOH (200 g/L), 5 g. picric acid	4.3198
103. Pb	180 c.c. NaOH (200 g/L)	0.0770
104. Pb	180 c.c. NaOH (200 g/L), 5 g. picric acid	1.3030
105. PbHg	180 c.c. NaOH (200 g/L)	0.0185
106. PbHg	180 c.c. NaOH (200 g/L), 5 g. picric acid	1.4685

In Table XIV the strength of the caustic solution was increased, the temperature raised, and picric acid was used as the oxidizing agent. The results are comparable to corrosion by acids. Amalgamation diminished corrosion slightly, and the oxidizing agent greatly stimulated corrosion of both amalgamated and pure metals. As in Table XIII, the stimulation of corrosion by the oxidizing agent is much less for lead than for zinc and tin.

TABLE XV.

Potentials of zinc, lead, and tin in normal NaOH and discharge potential of hydrogen on these metals.

	Poten.		Disch. P.	P. at 0.05 amp./dm.^{2}
Zn	+0.83?	<	+0.83	+0.96
Sn	+0.54	>	+0.65	+0.74
Pb	+0.21	>	+0.32	+0.40

Since gas was evolved from both commercial and C. P. zinc on immersing it in normal sodium hydrate, it is evident that the potential of this metal exceeds the discharge potential of hydrogen.

The values given for the discharge potential on tin and lead are probably slightly too low, for although bubbles of gas could be seen clinging to the cathodes, none was observed to escape. In every case an increase of current density caused a rise in potential of the cathode, and the limit was not reached at 0.05 amp./sq. dm.

What should be the effect of a vacuum on the rate of solution of amalgamated zinc by acids?

Carhart,[21] to show that the protection of zinc by amalgamation is due to the adhesion of a film of hydrogen, says "When amalgamated zinc is plunged in water, acidified with one-twentieth of its volume of sulphuric acid, it is not attacked at ordinary atmospheric pressure. But if a vacuum is produced above the liquid, bubbles of hydrogen are again freely evolved from the zinc surface. Upon readmission of the air, bubbles again adhere to the plate, and the chemical action is arrested."

If a vacuum stimulates corrosion of amalgamated zinc, it should also accelerate the dissolving of other metals in acids which corrode them but slowly at atmospheric pressure, because of the polarizing effect of hydrogen. To test this the experiments of Table XVI were tried, one set at atmospheric pressure, the other in such a vacuum as could be obtained by the laboratory filter pump.

TABLE XVI.

Temperature .....21 to 22° C.
Time .....45 hours
Area .....60 sq. cm.

Specimen	Reagents used	Loss in Grams
	(In Vacuum—2.4 to 3.5 cm. of mercury)	
107. Fe	N. H_2SO_4 190 c.c.	0.7332
108. Fe	N. H_2SO_4 190 c.c., 0.25 g. Na_3AsO_4	0.0083
109. Pb	N. CH_3COOH 190 c.c.	0.0374
110. Cu	N. H_2SO_4 190 c.c.	0.0044
111. Zn	N. NaOH 190 c.c.	0.0101
	(At Atmospheric Pressure)	
112. Fe	N. H_2SO_4 190 c.c.	1.6266
113. Fe	N. H_2SO_4 190 c.c., 0.25 g. Na_3AsO_4	0.0287
114. Pb	N. CH_3COOH 190 c.c.	0.0841
115. Cu	N. H_2SO_4 190 c.c.	0.0208
116. Zn	N. NaOH 190 c.c.	0.0772

At the start, the above test showed a much greater evolution of gas from the samples in the vacuum than from those under

[21] Primary Batteries, p. 34 (1891).

atmospheric pressure. Apparently Carhart's statement was true, and corrosion would be stimulated by the vacuum. Towards the end of the test, however, it was noticed that action was more rapid on several of the samples at ordinary pressure, but it was not then suspected that reducing the pressure had diminished, rather than increased, corrosion; it was only when the specimens were weighed that the truth was learned, *viz.*, that in every case reduction of pressure had diminished corrosion.

In order to ascertain that a possible difference in temperature between the two sets of specimens was not the cause of the different rates of corrosion, temperature measurements were made of solutions under similar conditions, except that no metals were present. Numerous bubbles of gas collected on the interior surfaces of the tumblers in the vacuum, which could only be air. The average of many readings showed a difference of less than four degrees in temperature, too slight to account for the lessened corrosion. The reason for the lessened corrosion in the vacuum is that the reduction of pressure removes from the liquids the dissolved air, which at atmospheric pressure acts as a depolarizer for hydrogen. Proof of the depolarizing action of the air present in ordinary solutions of the laboratory is seen by comparing the curves of cathode polarization in plates I and II of a paper presented before this Society in 1914 by C. N. Hitchcock.[22] The effect of removing the dissolved air either by boiling or by the use of a vacuum, was to cause a greater polarization of the cathode by hydrogen.

The experiment given by Carhart has been quoted in the same language by P. Benjamin,[23] and Tommasi,[24] and is attributed to De la Rive in 1843. The result of repeating it points to the desirability of testing with modern apparatus many of the alleged "facts" of our natural sciences that have been passed down to us through a long period of years.

This study of the corrosion of representative metals and of the electrochemical principles involved, has led to certain conclusions, some general, others specific.

[22] Trans. Am. Electrochemical Soc., **15**, 418 and 420.
[23] The Voltaic Cell, p. 347 (1893).
[24] Traité des Piles Electrique, p. 45 (1889).

1. The hypothesis of one of the authors, presented to this Society in a previous paper,[25] that the protective effect of arsenic on the corrosion of iron by sulphuric acid is due to polarization by hydrogen, is confirmed by the following facts:

(a) The corrosion by acids of iron protected by arsenic was invariably greatly stimulated by oxidizing agents.

(b) Arsenic does not protect iron from attack by corrosive agents which evolve no hydrogen by their action.

2. Amalgamation protects zinc from corrosion by acids because the discharge potential of hydrogen on mercury exceeds the potential of zinc. Protection by arsenic and by amalgamation are alike in their nature.

3. Tin and lead are only very slowly dissolved by non-oxidizing acids, although their potentials are such as would cause their ready solution in acids, if it were not for the unusually high discharge potential of hydrogen on them. Removal of hydrogen by an oxidizing agent causes these metals to dissolve readily in acids that otherwise corrode them very slightly.

4. Corrosion by acids, of metals below hydrogen in solution pressure or position in the usual electrochemical series, *viz.*, copper and silver, has been caused by the presence of oxidizing agents, and the reason for this action has been set forth in detail.

5. The oft-quoted statement that the corrosion of amalgamated zinc in dilute sulphuric acid is accelerated by a vacuum, has been shown to be incorrect. By removing the depolarizing oxygen of the air, reduction of pressure retards corrosion, not only of amalgamated zinc, but of other metals whose solution is hindered by a polarizing film of hydrogen.

6. As regards corrosion by acids the metals experimented with, and probably all others, may be classified as follows:

(a) Metals whose potentials exceed the discharge potential of hydrogen on them; these dissolve readily in acids, except such as form insoluble salts.

(b) Metals whose potentials are less than the discharge potential of hydrogen on them; these dissolve readily in acids only in the presence of oxidizing agents. Gold and platinum are not

[25] Trans. Am. Electrochemical Soc. (1912), 21, 340.

readily attacked by the acids in general, even when these contain oxidizing agents.

7. The superiority of nitric over other acids as a general solvent for the metals—long recognized—is due to its being at the same time an acid and an oxidizing agent, which enables it to dissolve metals of the second class, which non-oxidizing acids cannot do.

8. Oxygen is necessary to success in cyaniding gold ores because in dilute cyanide solutions gold is a metal of the second class.

9. Measurements of the discharge potential of hydrogen in solutions of potassium cyanide and sodium hydrate, and experiments on the corrosion of metals in the latter, lead the authors to think that the above classification of metals, the action of oxidizing agents, and protection by other metals, will apply to the *dissolving of any metal in any electrolyte* from which it displaces hydrogen when passing into solution.

10. The above classification of metals according to the relative magnitude of their potentials in comparison with the discharge potential of hydrogen on them, applies not only to the solution of metals, but to their electrolytic deposition. Plating baths for depositing metals of the first class cannot be strongly acidified without causing the deposition of much hydrogen in place of an equivalent amount of metal; but a large proportion of acid may be added to solutions for the deposition of metals of the second class without greatly lowering the current efficiency through the deposition of hydrogen.

Laboratory of Applied Electrochemistry,
University of Wisconsin.

DISCUSSION.

F. N. SPELLER[1]: I wrote out these notes relating to the practical application of the principles so well laid out in Watts' and Whipple's paper, for the reason that the subject of this discussion has already been partially covered in two or three rather extensive papers. It consists of the research on methods for conserving the life of galvanized-iron water pipe for hot water.

[1] Metallurgical Engineer, National Tube Co., Pittsburgh.

The rapid destruction of hot-water supply pipes under modern conditions presents a problem involving the principles discussed in this paper, the practical solution of which promises to be of far-reaching importance.

Water for domestic use is usually heated under 60 to 80 pounds pressure to about 160° F., and in large buildings is usually circulated in a closed circuit from the heater through the risers and returns. The large amount of water used in these days of convenient bathing facilities and the high temperatures carried has undoubtedly shortened the life of the pipe considerably.

Several years ago the writer started some experiments on the removal of the free oxygen from water by contact with a large surface of iron, and in December, 1915, a small plant, designed on this principle, was installed in this city, in the Irene Kaufmann Settlement. This is still in successful operation. (The method will be seen by looking at the section of the storage tank, filter, and heater shown on the following pages.)

By circulation of the heated water between steel sheets the latter become corroded, the free oxygen is used up primarily in keeping the surface of these plates active; the excess of hydrogen goes into solution.

The soluble gas collected from this water showed the following composition:

	Raw Water	Treated Water
Oxygen	15.6 percent	*1.59 percent
Hydrogen	0.3 percent	22.17 percent
Carbon Dioxide	0.3 percent	0.12 percent
Nitrogen (by Diff.)	81.8 percent	76.12 percent
Temperature	160° F.	162° F.

* The oxygen collected from the hot water is low, due to corrosion of the iron storage tank and connections.

The amount of free oxygen in domestic water usually ranges from 5 to 9 cc. per liter.

The system at the time this plant was installed consisted of old galvanized iron pipe, which was leaking every week and was ready to be replaced. No leaks have occurred with the "passive" water, and new pipe installed at the same time for test purposes shows practically no corrosion.

Brass or copper pipe naturally does not fail so quickly under

these conditions; however, it is to be expected from what we know of the electrochemical principles involved that these pipes may also be protected in the same way. In new installations of

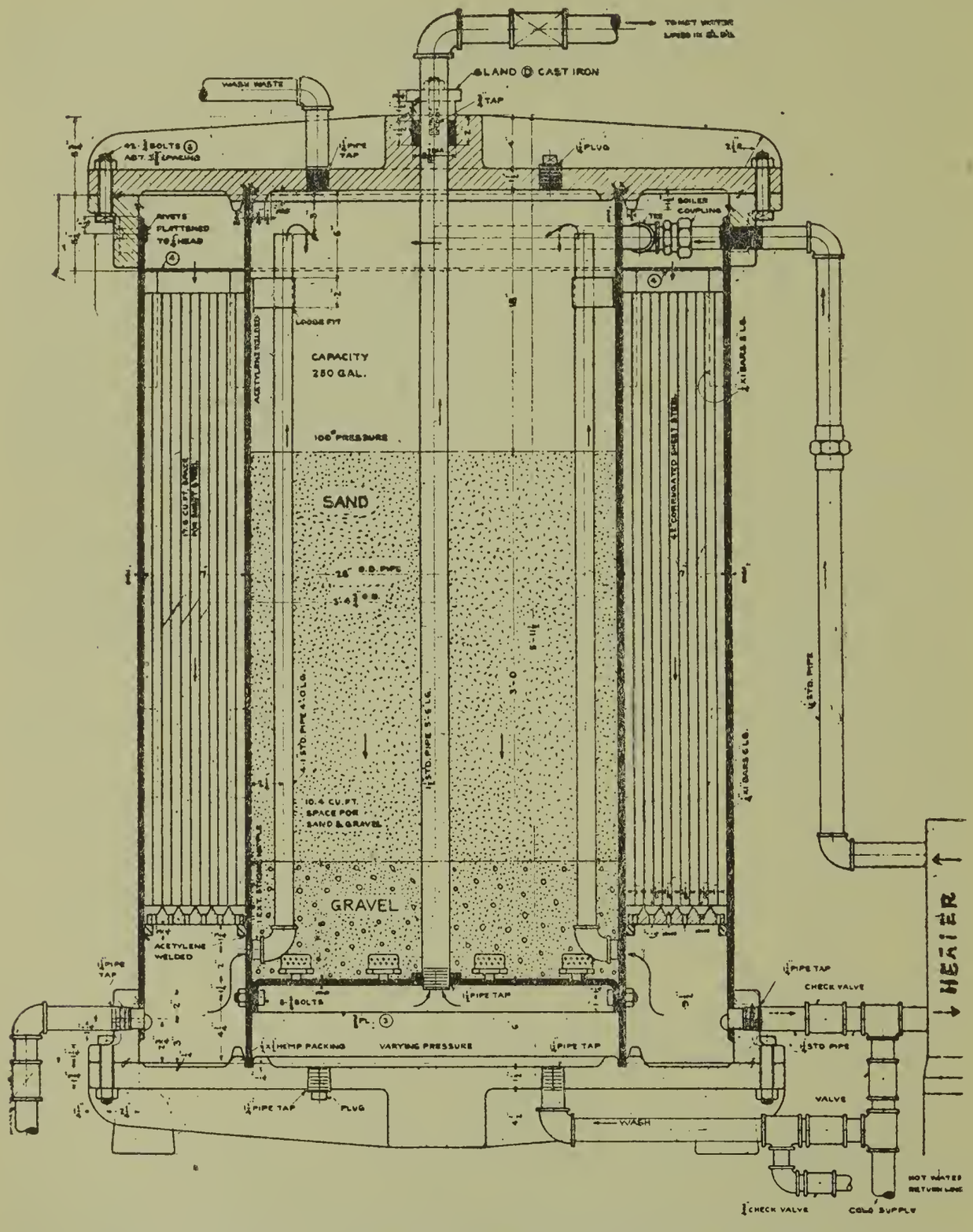

FIG. 1. System for Removing Dissolved Oxygen from Hot Water.

such a size as to warrant the extra equipment required to render the water passive to metal, galvanized steel pipe would seem to be the most economical material.

The unbroken film of hydrogen on the inside of a piping system of this kind is probably a more perfect protective coating than any other so far devised.

O. P. WATTS (*Communicated*): Further confirmation of the writer's views on the nature of the protection afforded by amalgamation has just been obtained with amalgamated iron. Since

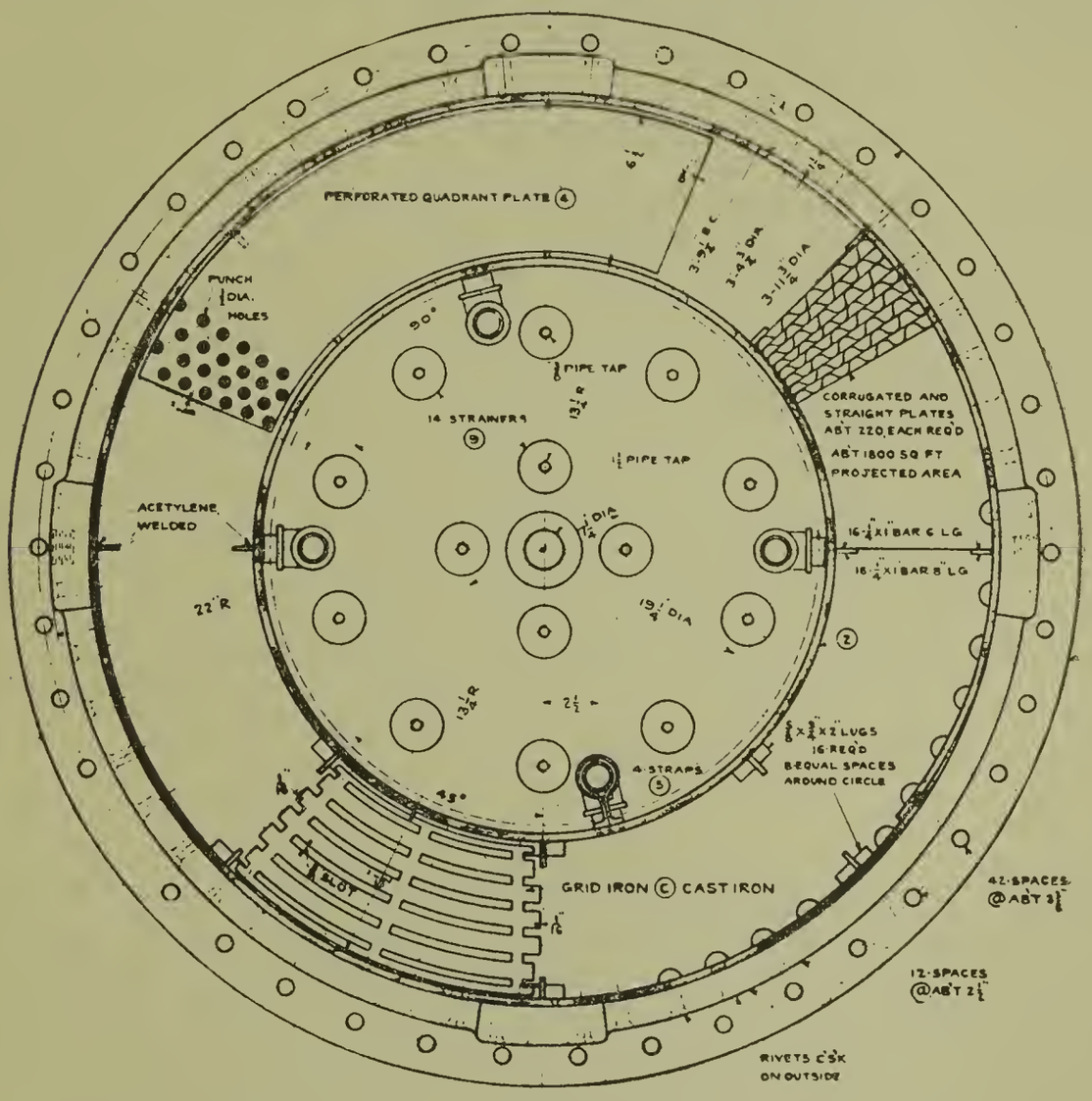

FIG. 2. Cross-Section of Fig. 1.

the discharge potential of hydrogen on mercury exceeds the potential of iron the theory leads to the conclusion that amalgamation should protect iron from corrosion in acids. By the use of sodium amalgam it has been possible to amalgamate iron. This was immersed in 15 percent sulphuric acid, and for two days there was no visible action, but on the third morning the iron had entirely disappeared and only mercury remained. The only explanation that the writer can offer for this sudden attack, where there had

previously been protection, is that the superiority of the force of cohesion between the particles of mercury over the adhesion between mercury and iron finally resulted in the gathering of the mercury into globules, thus leaving considerable areas of the iron exposed. Amalgamation protects iron from rusting in water for about the same length of time. It is hoped that further experiments will lead to a better understanding of this failure of amalgamation to protect iron for a longer time.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

THE CORROSION OF FENCE WIRE

By Oliver W. Storey.*

[Abstract.]

The importance of the problem and the difficulty of obtaining reliable data, are set forth. A large amount of new data obtained by the writer are given, with analyses of the wires and detailed descriptions of their condition. Many of the statements of the Government Farmers' Bulletin No. 239 are quoted and their accuracy discussed. A lengthy review is given of the history of the question of copper in steel and iron protecting it from corrosion, with the conclusion that the writer's conclusions are substantiated by numerous reliable observations, *viz.*, that copper is a strong protective against corrosion and that manganese is without influence either way.

About fifteen years ago the U. S. Department of Agriculture received many complaints from farmers stating that wire fences corroded rapidly. As a result of these complaints an investigation was begun at that time by the U. S. Department of Agriculture to bring about, if possible, an improvement in the corrosion-resisting qualities of fence wire. Notwithstanding this investigation, the preliminary results of which are embodied in Farmers' Bulletin No. 239,[1] there appears to have been made but slightly better wire since that time. Farmers continue to complain of the short life of the present-day wire as compared to the older wires, some of which are still in service. This paper gives the results of an investigation which has determined the cause of the longevity of the older steel wires.

* Metallurgical Engineer, C. F. Burgess Laboratories, Madison, Wis.

[1] See references collected at end of paper.

A study of the analyses of a large number of fence wires has resulted in the conclusion that the presence of a sufficient amount of copper in steel wire determines whether that wire is resistant to rapid corrosion. The results have without exception shown that where two steel wires have been subjected to identical corroding conditions the wire with an appreciable percentage of copper has shown the lesser corrosion. In the light of recent extensive investigations on the corrosion of iron and steel, especially copper steels, these results, covering a corroding period of from 20 to 30 or more years, are of much significance in that they confirm the results obtained in corrosion tests covering a short period of time.

The general complaint of the users of wire fences, given in Farmers' Bulletin No. 239, was that the recently manufactured fence wire corroded rapidly, lasting but a few years, while some of the old wire which had been erected on their farms thirty years before was still in service. As a result of these complaints A. S. Cushman[1] made an investigation in which he came to the following conclusions:

"1. That modern Bessemer and open-hearth steel rusts much more rapidly than iron wire.

"2. That manganese, especially if it is unevenly distributed in the steel, is at least in part the cause of the trouble."

Since that time Cushman has been an advocate of low-manganese pure irons wherever resistance to corrosion is desired and recently has also contended that copper does not increase their resistance to corrosion.

Whether Cushman was justified in concluding that the absence of manganese and high purity caused high resistance to corrosion will be more fully discussed.

OBTAINING OF RELIABLE DATA.

In making a study of corroded fence wire it is difficult to obtain reliable data on the length of service of wire and it also is difficult to obtain comparative specimens which have been subjected to identical corroding conditions. Where fences have been erected for more than a few years it is almost impossible to obtain an accurate history of them. In the case of barbed wires, strands in good condition are usually salvaged from the old fence which is

being replaced and this adds to the complications. Therefore, in comparing different strands of barbed wire great care must be used to determine the history of each strand.

However, it is possible in some cases to approximate the age of fence wire, especially wire that has been erected for a long period. Occasionally wire fences are nailed to trees and the depth to which the wires are imbedded in the growing tree gives a basis for estimating their age. Much of the barbed wire erected 20 to 30 or more years ago had barbs of peculiar patterns that were made only during that period. The age of these wires may be approximated.

The rate of corrosion of iron and steel varies with the location. Not only does this hold true with reference to conditions as influenced by the topography of the region and the presence of industrial corroding gases, but also to the position of the wire on the fence; whether it is the bottom or top strand, whether shaded, covered with brush, next to a post or tree, or in the open.

A careful inspection of a wire fence shows that the bottom strands generally are in much better condition than the top strands. This is also true of the wire lying next to a post or a tree and also where the wire is covered or protected by trees and shrubbery. Since the different strands of wire on the same fence are subject to varying corrosive action, great care must be used in comparing them.

The nearest to the ideal condition for a corrosion test is found in a strand of barbed wire made by twisting together two wires of different compositions. Even here the galvanizing on one wire may be slightly different from the other. While this influences a short-time corrosion test, it is not of moment in a test covering 25 or 30 years, provided that the galvanizing is of the usual grade found on barbed wire.

The objection will also be raised that electrolytic action between the two wires will influence the results appreciably. A careful study has shown that the influence of electrolytic action on the corrosion of two dissimilar wires in a strand of barbed wire is practically nil. There are few points of contact between the two wires and the potential difference between the two wires is necessarily minute. The work of Aston,[2] together with some research by C. F. Burgess and also by the writer along the same lines,

indicates that the electrolytic action of rust on the surface of iron and steel is far more important than any minute difference in potential due to slight differences in composition. Many samples were found where the two wires in a strand of barbed wire were not in contact over a distance of an inch or more, but the corrosion at these places did not vary from the points where the wires were in contact, though the two wires were dissimilar in composition and corroded at different rates. These observations led to the conclusion that the two wires of a strand of barbed wire that has been in service for 20 to 30 years may be compared and conclusions based on any difference in composition.

DATA ON FENCE WIRE.

At Mineral Point, Wisconsin, and at several other points in the same State, strands of barbed wire were found in which the two wires were corroding at a dissimilar rate. In some cases the difference in corrosion was slight while in one case one wire had practically corroded entirely away while the other was but slightly corroded. Fortunately the age of most of these wires could be approximated.

Much of the barbed wire made previous to 1892 was made by small manufacturers who did not control the sources of supply of raw material, but merely bought the wire and fabricated the strands. Under such conditions it is not surprising that many strands of barbed wire were built up of two wires that corroded at unequal rates.

Many of the fences, when erected, had been fastened to oak trees. These barbed wires, in some instances, are now embedded to a depth of four inches. The barbs are mostly of the obsolete two-point Lyman, and other similar patterns which were generally discontinued at the expiration of the Glidden patents in 1892. Many of the barbed wires obtained were probably erected in the period from 1885 to 1892 or 25 to 30 or more years ago.

Not all of the barbed wire was of the type in which one wire corroded more rapidly than the other. This was a characteristic of the older wires. Many specimens were obtained in which the two wires corroded at equal rates. The barbed wires of more

recent manufacture generally do not show any difference in the rate of corrosion between the two wires composing the strand.

A close examination of barbed wire fences at various places in southern Wisconsin shows that there is available much wire in which the two wires composing the strand corrode at dissimilar rates. A close inspection is necessary to note this difference, since the twisted wires confuse the eye. Often it is necessary to untwist a small section and flake off the adhering rust.

One old woven wire fence showed a difference in the rate of corrosion of the various horizontal wires. Care has to be taken in selecting woven wire specimens, since most woven fencing is of more or less recent origin and there is a decided difference in the durability of the galvanizing on the various horizontal wires. The top strands also corrode at a much higher rate than do the lower strands in the same fence.

The appearance of the rust varies depending upon the locality, and upon the resistance of the specimen to corrosion. Where the atmosphere is relatively pure and the corrosion rate low, the rust on all fence wire, after several years' exposure, is a deep brown in color and adheres tightly to the wire. When the iron is first exposed to the atmosphere, after removal of the galvanizing or adhering rust, the rust is bright red in color. The color appears to change gradually to a deep brown.

Wherever the atmosphere becomes more corrosive, due not only to the presence of industrial gases, but also to excessive moisture as in river valleys and swamps, and during the moist warm spring and summer months, there is a marked difference between those wires that are highly resistant to corrosion and those that are easily corroded. The rust on those wires that resist corrosion is a deep brown and adherent, while the rust on those wires that show small resistance to corrosion is yellow or light red in color, is flaky and may be easily removed. Also, the less resistant wires are deeply pitted, while the more resistant wires corrode more or less uniformly.

Complete analyses of several strands of barbed wire were made when an investigation with reference to the damage done by sulphur gases at Mineral Point, Wisconsin, was first begun. It was necessary to determine the kind of steel used and also to determine if possible the cause of the marked difference in corro-

sion of the wires constituting the strands of barbed wire. These total analyses are given in Table I.

A study of these analyses shows that there is no relation between the percentage of any element and the amount of corrosion with the exception of copper. These analyses also show that all of the barbed wire is made of Bessemer steel, excepting one which is made of wrought iron. The woven wire fence (Pittsburgh Welded Steel) is made of basic open-hearth steel.

Many different kinds of barbed wire have been examined, but only one made of wrought iron wire has been found.

In view of the work of Burgess[3] and Buck[4] and various other investigators who have shown that copper when present in small amounts in iron and steel decreases the rate of atmospheric corrosion to a marked extent, it seemed certain that the copper was the cause of the high resistance to corrosion of the wires that had been analyzed. Therefore, the copper content of a large number of wires used in barbed wire fences was subsequently determined. The results are given in Table I.

EXPLANATORY DATA AND DISCUSSION OF TABLE I.

Samples from Mineral Point, Wisconsin.

No. 1.—Pittsburgh welded steel fence. The horizontal wires corroding at different rates. Location of wire on fence did not influence rate of corrosion. The vertical wires are light gage and badly corroded. The wires high in copper are in best condition. (1a and 1b).

No. 2.—About five strands of old 2-point Lyman barbed wire found on an old gate. Some strands are in a good state of preservation while others are badly corroded. It is probable that these five strands were erected at the same time. Good strand contains copper. (2b). (See group No. 1 in photograph. Good strand on left, poor on right.)

No. 3.—An old fence composed of two old 2-point Lyman barbed wire strands embedded in burr oaks to a depth of four inches at some places. One strand in good condition and the other at same place in poor condition. The good wire (3a) contains copper. See No. 5. (Good strand is shown on right in group No. 6 in photograph.)

TABLE I.

Analyses of Fence Wires.

Location	No.	Kind of Metal	Cu	C	Mn	P	S	Si
Mineral Pt.	1a	B. O. H.	0.18	0.07	0.42	0.021	0.055	0.03
" "	1b	Steel	0.087	....	....	....	....	....
" "	1c	Steel	0.041	....	....	....	....	....
" "	1d	Steel	Tr.	....	....	....	....	....
" "	2a	Bess.	0.017	0.05	0.45	0.077	0.072	....
" "	2b	Steel	0.16	....	....	....	....	....
" "	3a	Bess.	0.22	0.10	0.31	0.090	0.069	0.13
" "	3b	Steel	0.018	....	....	....	....	....
" "	4a	Bess.	0.11	0.06	0.49	0.098	0.120	Tr.
" "	5a	W. I.	nil	0.03	0.10	0.136	0.010	....
" "	6a	Bess.	nil	....	0.25	0.060	....	....
" "	6b	Bess.	nil	0.07	0.29	0.043	0.085	....
" "	6c	Bess.	0.15	0.09	0.64	0.152	0.048	....
" "	7a	Bess.	0.023	0.13	0.62	0.114	0.066	0.01
" "	8a	Bess.	nil	0.05	0.25	0.129	0.098	....
" "	8b	Bess.	0.24	0.07	0.45	0.116	0.097	....
" "	9a	Steel	0.038	....	....	....	....	....
" "	9b	Steel	0.088	....	....	....	....	....
" "	10a	Steel	Tr.	....	....	....	....	....
" "	10b	Steel	0.06	....	....	....	....	....
" "	11a	Steel	0.16	....	....	....	....	....
" "	11b	Steel	0.097	....	....	....	....	....
" "	12a	Steel	0.120	....	....	....	....	....
" "	13a	Steel	0.397	....	....	....	....	....
" "	14a	Steel	0.11	....	....	....	....	....
" "	14b	Steel	0.04	....	....	....	....	....
" "	15a	Steel	0.06	....	....	....	....	....
" "	15b	Steel	0.224	....	....	....	....	....
" "	About 15 to 20 barbed wires, both new and old and heavily corroded, contained less than 0.03% Cu.							
Cuba City	16a	Steel	0.07	....	....	....	....	....
" "	16b	Steel	0.13	....	....	....	....	....
" "	Ten barbed wires, both new and old and heavily corroded, contained less than 0.02% Cu.							
Madison	17a	Steel	0.15	....	....	....	....	....
"	17b	Steel	0.097	....	....	....	....	....
"	18a	W. I.	0.01	....	....	....	....	....
"	18b	Steel	0.063	....	....	....	....	....
"	19a	Steel	0.031	....	....	....	....	....
"	19b	Steel	0.015	....	....	....	....	....
"	20a	Steel	0.130	....	....	....	....	....
"	20b	Steel	0.008	....	....	....	....	....
E. Milwaukee	21a	Steel	0.148	....	....	....	....	....
"	21b	Steel	0.005	....	....	....	....	....
"	22a	Steel	0.233	....	....	....	....	....
"	22b	Steel	0.005	....	....	....	....	....
"	23a	Steel	0.01	....	....	....	....	....

No. 4.—An old Baker barbed wire in good condition. Next to it is a recently erected barbed wire in poor condition. Old wire contains copper.

No. 5.—Wrought iron barbed wire in good condition except for a few deep pits. This wire was up more than 25 years, according to the owner. Found near No. 3; both embedded about four inches in burr oaks. Free from copper, low in manganese, but also very low in sulphur. Much slag and wire brittle. (Strand on left in group No. 6 in photograph.)

No. 6.—In this sample one wire (6a and 6b) is entirely corroded away in places, while the other is in good condition (6c). Most striking of all samples. Barbed wire of old 2-point Lyman pattern. Good wire contains copper and is high in manganese. Poor wire low in manganese and free from copper. Fence in moist, swampy meadow. (See wire No. 4 in photograph.)

No. 7.—New 2-point Glidden wire erected about seven or eight years ago. Entirely corroded away in places. Near No. 6. Low copper.

No. 8.—Old 2-point Lyman barbed wire subjected to locomotive and some smelter fumes. Fence posts nearly rotted away. Wire low in copper (8a) badly corroded, while wire high in copper (8b) is in good condition. Good wire also higher in manganese, and other elements about the same in each.

No. 9.—Old 2-point Lyman barbed wire. Wire (9a) low in copper. Corroded more than wire (9b) high in copper.

No. 10.—Old 2-point Lyman barbed wire. Wire (10a) low in copper corroded much more than wire (10b) containing copper.

No. 11.—Old 2-point Lyman barbed wire. Slight difference in rate of corrosion. Both in good condition. High copper wire (11a) the better of the two. Complete analysis would probably show up difference in other elements as above percentages of copper should not make difference in corrosion rate.

No. 12.—Old 2-point Lyman barbed wire from scrap heap. Old fence destroyed by floods. New fence up about five years. Nos. 12 and 13 in good condition, while new fence almost ready to fall in pieces. Wire in low, damp location and subjected to locomotive and smelter fumes. Nos. 12 and 13 contain copper, while newer

fence wire contains less than 0.02 percent. (See group No. 3 in photograph. Wire on left is No. 13, while that on right is badly corroded newer wire.)

No. 13.—Old 4-point Glidden barb. See No. 12.

No. 14.—Old peculiar spread barb, made about 30 years ago. In same swampy location as No. 6. Wire in good condition (14a), contains larger amount of copper than wire in poorer condition (14b). (Same pattern as wire on right in group No. 5 in photograph.)

No. 15.—Old 2-point Lyman barb in good condition. Subject to some smelter fumes. Imbedded several inches in burr oak. Both wires contain copper.

Numerous samples of both older and newer barbed wire that were badly corroded were analyzed for copper. These samples came from about the same locations that the numbered samples were obtained. None of these samples contained more than 0.03 percent copper and most of them were under 0.02 percent. None of the newer barbed wires contained more than 0.02 percent copper and none showed high resistance to corrosion.

Samples from near Cuba City, Wisconsin.

A number of samples of barbed wire were obtained from the immediate neighborhood of an ore roaster near Cuba City, Wisconsin. The sulphur dioxide gases are causing rapid corrosion of all fence wire within 400 yards of the stack. The smelter had been in operation about four years when the samples were taken.

No. 16.—About 250 yards west of the stack one barbed wire of the old 2-point Lyman pattern was found in excellent condition, while the fences in the immediate neighborhood were badly corroded. These fences were mostly of more recent erection, though one or two old wires were found. The rust on this well-preserved wire is deep brown as compared with light brown on the heavily corroded specimens. The heavily corroded specimens contain less than 0.02 percent of copper, while the good strand was composed of two wires containing 0.07 and 0.13 percent respectively. There is no difference in the corrosion rate between the two wires. (Wire on left in group No. 2 in photograph is good strand, while that on right is badly corroded wire from same place.)

Samples from Madison, Wisconsin.

No. 17.—Old 2-point Lyman barbed wire, found on Eagle Heights. Old "line" fence. Posts nearly rotted away. Wire in excellent shape. Both wires contain copper.

No. 18.—Old barbed wire found on Regent Street. Same make as No. 5. Both wires in excellent condition. Appears to have been painted originally. One wrought iron and the other of steel containing copper and show no difference. Evidently copper steel as good as wrought iron. See No. 19.

No. 19.—Old peculiar looped barb, on same fence with No. 18, but in poor condition. Both wires low in copper.

No. 20.—Old peculiar looped barb. On right-of-way of spur of Illinois Central Railroad. Practically no traffic. Wire high in copper (20a), in good condition, while other (20b) low in copper, is in much poorer condition.

Samples from East Milwaukee, Wisconsin.

These samples were obtained at the northeastern city limits of Milwaukee, where the atmosphere is not contaminated with smoke and the corrosion is about normal.

No. 21.—Old 4-point Lyman barbed wire. Wire salvaged from old fence and used to reconstruct fence 11 years ago. Good wire (21a) contains copper, while poor wire (21b) contains a trace. (Strand on left in group No. 5 in photograph. Note difference between the two wires.)

No. 22.—Old spread barbed wire same as No. 14. Wire salvaged from old fence and used to reconstruct fence 11 years ago. Good wire (22a) contains copper, while poor wire (22b) contains trace. (Strand on right in group No. 5 in photograph. Note difference between the two wires.)

No. 23.—New barbed wire was used to construct a fence across alley from No. 21 and 22 about seven years ago. This wire is in poor condition now. Practically free from copper.

CONCLUSIONS.

The data obtained show conclusively that copper steel is highly resistant to corrosion and that the older steel fence wire containing copper is much more durable than that free from copper. The resistance to corrosion is independent of the percentage of manganese in the steel.

DISCUSSION OF FARMERS' BULLETIN NO. 239.

Unfortunately Cushman did not publish the detailed data on which he based his conclusions, that manganese caused the rapid corrosion of fence wire, though he found that many samples high in manganese were highly resistant to corrosion. On page 15 he states:

"It is sufficient for our present purpose to state that it soon became noticeable that the majority of the old wires sent in which were in good condition were either free from manganese or contained only very small amounts—0.2 percent or under—of this metal. It is true that many of the good wires ran as high as 0.5 percent and even higher in manganese, but the fact was nevertheless noticeable that the bad wires, with very few exceptions, contained manganese, while the good wires were frequently if not always free from it."

This last sentence is more or less contradictory, but evidently the manganese was not the cause of the trouble if many of the good wires ran as high as 0.5 percent and even higher in manganese and some of the bad wires were free from it.

The most important element—copper—was not determined.

Although the bulletin is not clear on this point, apparently wrought iron and steel, both open-hearth and Bessemer, are compared directly by chemical analysis, and conclusions based on this comparison. There are "steels and steels" and the same expression may be applied to wrought iron. It is evident that some of the steel fence wire was highly resistant to corrosion. A direct comparison between good and poor wrought iron and especially good and poor steel would have been of more value, since practically all fence wire is made of steel. Wrought iron is entirely different from steel physically, so that much care must be used in basing any opinions founded on the difference in the chemical analyses of these two materials, especially when all of the elements present have not been determined.

On page 21 of the bulletin the dissimilarity between steel and wrought iron and between different steels is described:

"While it is true that puddled iron is in a large measure protected from corrosion by the presence in its fibres of mill cinder, this has nothing to do with the fact that in almost all modern steel woven-wire fences some wires will be found to far outlast others, independent of the original weight of the galvanized covering which they carry. If in woven wire fence all the wires last as well as the best ones do, there would have been no complaints, and this investigation would never have become necessary."

The heterogeneous structure of puddled iron is described at the top of page 13:

"By this means the impurities are oxidized and burnt off, and the slag or cinder which always forms is worked into the metal, so that when it comes to be rolled not only are the impurities very evenly distributed throughout the mass, but it possesses the structure of a bundle of fibres, each one of which is coated with a film of cinder, which protects it in very large measure from subsequent rusting."

Wrought iron contains from 0.5 to 2 percent of this slag, but this is not shown in the usual analyses. Following are typical analyses of wrought iron, basic open-hearth and Bessemer steel fence wire taken from Table I:

TABLE II.

	Carbon	Manganese	Phosphorus	Sulphur	Silicon
Wrought iron.....	0.03	0.10	0.136	0.010	....
Basic open-hearth....	0.07	0.42	0.021	0.055	0.03
Bessemer.....	0.07	0.45	0.116	0.097	...

The above analyses show the wrought iron to be low in manganese as compared to the steel, but it should also be noted that the sulphur content is about 10 to 20 percent of that of the steel. Buck[4] has conclusively shown that sulphur affects the resistance of steel to corrosion to a marked degree. The low sulphur content of wrought iron should make it more resistant to corrosion as compared to the steels containing from 5 to 10 times as much. Investigation has not yet shown that manganese decreases the resistance of steel or iron to corrosion.

Cushman concludes that most of the old iron wire erected from 1875 to 1885, and before that period, is much more resistant to corrosion than modern steel. It will later be shown that the iron ores used during and previous to that period contained copper in varying amounts. Since practically all of the wrought iron was made in the eastern part of the United States, where most of the ore was obtained at that time, it is probable that much of the wrought iron contained copper. Since the copper was not determined in the iron or steel examined by Cushman, this point is open to further investigation.

FIG. 1.

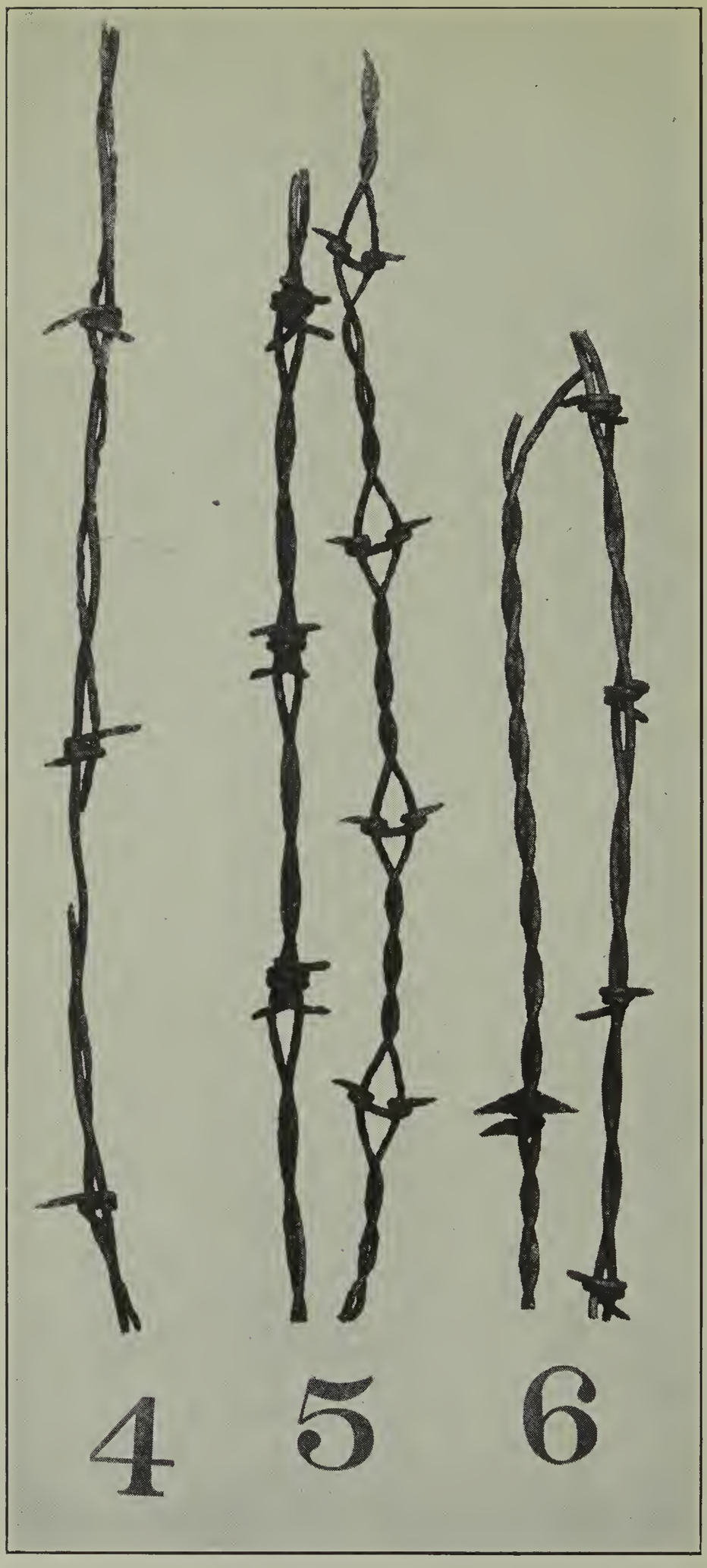

FIG. 2.

Unfortunately, only two wrought iron barbed-wire specimens, both of the same make, were found. With such meager data it is impossible to make any comparisons as to the durability of various wrought irons. However, one of the strands, No. 18, was composed of a wrought iron and a steel wire, the latter containing 0.06 percent copper. The steel and wrought iron wires were both in excellent condition, showing little corrosion, while a steel barbed wire, No. 19, which was next to it and of obsolete pattern, was in poor condition. This one comparison indicates that copper steel is as durable as wrought iron.

The bulletin does not define what is meant by "Corrosion of Fence Wire."

There are two steps in the corrosion of galvanized wire—the first resulting in the destruction of the galvanizing, and the second resulting in the destruction of the iron or steel base.. Usually a fence is not considered useless when rust begins to appear, but rather when the iron or steel base has corroded so badly that it can be of no further service. Generally the first period is short compared with the second.

A heavy coating of zinc of good quality will insure a long life for the wire, even though the iron base is of poor quality. In this case the wire will not become rusty and have a poor appearance as long as the galvanizing is intact. On the other hand, if a rust-resisting grade of steel wire is covered with a thin coating of low-grade galvanizing, the life of the wire may be as long as in the first case, and probably longer, though the wire will become rusty in two or three years.

Since galvanized barbed wire usually receives but a thin coating of zinc, the life of the wire depends principally upon the life of the iron or steel base.

If a fence wire is supposed to have failed when rust appears, this cannot be regarded as a measure of the corrosion resistance of the steel, but, instead, of the galvanizing. The steel or iron base has little influence except as it affects the quality of the galvanizing. Little is known of the effect of the iron or steel base on the quality of the galvanizing and this is a subject worthy of extended research.

Throughout the bulletin this failure to keep the durability of the

galvanizing separate from that of the iron or steel base has resulted in confusion and given erroneous impressions to the reader.

The following extracts from Bulletin 239, page 21, illustrate this point:

"In one fence which has been under observation for four years one wire was in perfect condition, although it carried a light covering of zinc, while the wire next to it was badly rusted from end to end. Careful chemical analyses were made of these two wires, and, in order to check the results secured, samples were sent to one of the most eminent iron chemists in the country. This chemist reported as follows:

"'We have examined these samples, finding as follows:

Constituents	Good Wire, Percent	Bad Wire, Percent
Carbon	0.17	0.17
Manganese	0.45	0.53
Phosphorus	0.092	0.096
Silicon	0.070	0.060
Sulphur	0.059	0.083

You will note that, so far as these two samples go, there is very little difference in the wire, and practically no explanation chemically as to why one should be good and the other bad. They might almost be from consecutive heats from the same Bessemer converter. I do not think they are from the same heat. Notwithstanding this similarity of analysis of the samples which we have examined, it is more than probable that there may be quite unequal distribution of the manganese in the two samples.'"

While the statement is not specific, it is supposed that "perfect condition" means that the zinc coating is intact. It is difficult to see how a comparison of the analyses of the steels in different wires justifies any conclusions with reference to the durability of the wires and the causes of corrosion when the steel of one wire has not corroded and the galvanizing is in perfect condition. It would seem to show that there was a decided difference in the quality of the galvanizing.

Page 23—"A very much better covering and perhaps a more durable wire can be made by what is known as the double galvanizing process. The double process does not, as its name implies, mean that more than one coating of zinc is put on, but only that about twice as much zinc by weight is carried by the wire. In the double process the wire is drawn much more slowly through the zinc bath and does not pass through asbestos wipers, but is smoothed by passing through a shallow bed of slightly damp charcoal powder. Telegraph wire is usually treated by this method and is generally acknowledged to be more durable than fence wire, but it is worth noting at the same time that care is exercised in the manufacture of telegraph wire to keep the manganese low, because the presence of this element increases the electrical resistance of the wire."

Observation shows that the galvanizing on telegraph wire is much more durable than that on fence wire. The double-weight

of zinc used gives more than a two-fold increase in the life of the galvanizing. The cause of the increased life is then not due to the low manganese content of the steel base, but to the heavy coat of galvanizing, which is made possible by the large gage wire generally used.

Page 24—"Although, as has been said in an earlier paragraph, it is not the intention to maintain that the weight of the galvanized coating has nothing to do with the lasting quality of the fence wire, in view of the evidence which has been collected, it is impossible to believe, as many people do believe, that insufficient galvanizing is the whole cause of the difficulty. Among the wire fences that have been under the careful observation of the writer for a long time is one woven-wire fence five years old that contains among its twelve horizontal wires, all of one roll, seven wires that are perfectly good from beginning to end, four that are partially rusted, and one that is badly rusted, without a particle of zinc remaining on it. Of the seven perfectly good wires, six are on the bottom. Now, if the theory of electrolysis is left out of the question it would seem that the good wires received a better coating of zinc than the bad wires. This explanation is not supported either by observation or experiment. Two wires are made in the same mill; pass through the same zinc bath; are wiped off in precisely the same way; chemical analysis shows them to have practically the same weight of zinc covering per pound of metal; and yet one wire will outlast the other 5 to 1 on the fence. It is, however, entirely possible that the perfection with which the manganese and other impurities are distributed may happen to be more perfect in one ingot than it is in another. Within certain practical and possible limits it is probably true that, other things being equal, the wire with the heavier zinc coating per unit of surface area will be the most resistant to weather conditions."

In this case the writer again maintains that the quality of the galvanizing determines how quickly the wire becomes covered with rust. The galvanizing on the same wire varies irregularly along its entire length, as may readily be seen on any woven wire fence that is beginning to rust. It is hardly probable that galvanizing of the same quality will be found in all of the strands of any woven wire fence. Also the weight of the zinc per unit of area does not alone determine the quality. If the galvanizing varies in quality it is difficult to see how any conclusions, which are based on the relative durability of the galvanizing, may be made as to the corrosion-resisting qualities of the iron or steel base, especially before the zinc has been removed.

However, the composition of the iron and steel does become of the utmost importance after the removal of the galvanizing by weathering and usually the durability of the iron or steel base plays a more important part in the life of the fence than the galvanizing.

REVIEW OF COPPER STEEL INVESTIGATIONS.

According to Friend,[5] Karsten (British Association Reports 1838, p. 265) was the first to class the copper alloys along with those less liable to corrode than ordinary iron. He then states that the early researches showed that copper irons were poorer than ordinary iron, but later it was found that the sulphur present with the copper caused the trouble. The reputation of the copper steels in the physical tests also suffered, owing to the accompanying sulphur that was so often present in excessive amounts.

The first recorded research work on the corrosion of copper steels is that of Williams.[6] In 1900 he made some atmospheric corrosion tests of steels with a varying content of copper, and also of wrought iron with and without copper. The results follow:

Bessemer Steel Series.

	Percent Copper	Percent Loss
A. Soft Bessemer	....	1.85
B. " "	0.078	0.89
C. " "	0.145	0.75
D. " "	0.263	0.74

Steel and Wrought Iron Series.

	Percent Loss
Soft Bessemer	1.65
Wrought iron, No. 1	0.76
" " " 2	0.80
" " " 3	0.87
" " " 4 (0.393 Cu)	0.53

The results obtained with the steel, check the results of Buck closely, in which he found that 0.05 percent of copper gives almost a maximum protective effect.

Williams' work was quickly followed in 1901 by that of Stead and Wigham,[7] who were studying the effect of copper in steel on wire-drawing. A series of 14-gauge wires were left in flowing Middlesborough town water for nine weeks. Similar samples were exposed to the salt water in the tidal River Lees at Middles-

borough. While Cushman[8] states that the results were confusing, Stead and Wigham conclude, "It may be considered as proved that copper in iron and steel retards corrosion."

Following is a table showing the analyses of the steels used by Stead and Wigham and the percentage loss:

Bessemer Rail Steel.

		C	Mn	Si	S	P	Cu	Percent Loss
Series I	A...	0.330	1.090	0.028	0.118	0.049	0.012	19.0
	B...	0.320	0.640	0.028	0.055	0.048	1.286	11.5
Series II	C...	0.480	0.933	0.070	0.047	0.084	0.033	12.0
	D...	0.490	0.933	0.075	0.047	0.082	0.889	13.5
Series III	E...	0.310	0.676	0.084	0.047	0.084	0.012	14.7
	F...	0.200	0.676	0.084	0.044	0.082	2.000	4.7
Series IV	G...	0.340	0.940	0.065	0.030	0.063	0.025	10.6
	J...	0.420	0.930	0.065	0.030	0.067	0.480	8.9

Crucible Steel.

Series V	N...	0.990	0.463	0.168	0.034	0.021	0.010	12.7
	X...	0.970	0.491	0.177	0.034	0.023	0.460	10.1

A study of the results shows that copper decreases the corrosion of steels. The only exception is in Series II, where the low copper steel has a slight advantage over the high copper steel, though it may be pointed out that the former contains over 0.03 percent of copper, at which point Buck has shown that the influence of copper is noticeable. Manganese does not appear to influence the amount of corrosion, while the high sulphur content of the first steel probably accounts for its excessive corrosion.

In 1912, Burgess[3] found that when even small amounts of copper were added to electrolytic iron, the atmospheric corrosion was lessened greatly.

This work was closely followed by that of Buck,[4] whose important researches on a commercial scale have established the superiority of copper steel over the other steels, and have also

established the limits in which this high resistance to corrosion may be secured. Incidentally the action of various impurities in steel is being determined.

ORIGIN OF COPPER IN STEEL FENCE WIRE.

The data presented show that much of the Bessemer steel made 25 or 30 years ago contained copper, while most of that made more recently, and at the present time, contains but a trace. The presence of copper in open-hearth steels is easily explained, as it comes largely from the copper contained in the scrap iron which is used in that process. Since metallic copper was not purposely added to the Bessemer steel made 25 or 30 years ago, it must have had its origin in the iron ore used in producing the pig iron.

A study of the ores of the United States shows that the Lake Superior ores produce a steel containing at most but a trace of copper. However, the ores obtained in the eastern United States contain varying amounts of copper, the Cornwall ores of eastern Pennsylvania sometimes producing an iron or steel analyzing as high as 1.00 percent copper. Some of these ores are treated for their copper content.

About 25 to 30 years ago the Lake Superior ore district had not assumed the important part in the metallurgy of iron and steel that it now occupies, while the iron ores of the eastern United States formed an important part of the output of this country. In addition to this, 200,000 to 300,000 tons of copper-bearing iron ores were imported in some years from Cuba.

The following extract from H. H. Campbell's book on the "Manufacture and Properties of Structural Steel," published in 1896, shows that the older steels and wrought irons usually contained copper:

"Influence of Copper—The iron made from the ores of Cornwall, Pa., contains from 0.75 to 1.00 percent of copper, and large quantities of rails have been made from this iron alone, but it has oftener been the custom at Eastern steel works to use from 25 to 50 percent of this iron in the mixture, the rest being made from foreign ores. Other deposits contain considerable quantities of this element, notably some beds in Virginia, while the ores of Cuba give an iron with about 0.10 percent of copper.

"Not only has such metal been put into rails, but into all kinds of steel, both hard and soft, and large quantities have been worked in puddle furnaces and in foundries, so that the miscellaneous cast-iron, wrought-iron, and steel scrap, throughout the East, is very apt to contain quite an appre-

ciable quantity of copper, and as steelmakers will thus forever have this element to handle it is of pressing importance that its effect be understood. The necessity for such knowledge is the more marked, as it is the custom in certain favored districts to intimate that copper is injurious, although definite proof is always lacking.

"Most of the Bessemer steels which are recorded in this book contain from 0.30 to 0.50 percent of copper, while much of the open-hearth steel is of the same character, and this will be sufficient proof that the best of steel may contain a considerable proportion. If, therefore, it appears from a set of experiments that copper exerts a bad effect, then one of two things follows:

"(1) That the experiments have left some factor out of the question.

"(2) That the maker of good steel has some trick by which he overcomes the enemy.

"It would be a cause for satisfaction if we could boast that the latter supposition were true, but, as a matter of fact, we have never known that copper injured the cold properties of steel in any way, and it is unnecessary to add that no system has been devised to obviate its influence.

"Hard and soft steels of our manufacture have found their way into all channels of trade, and, although many failures have come, as they have everywhere, from high carbon, high manganese, or high phosphorus, there have been no cases where it was necessary to invoke the aid of copper. This fact outranks and transcends in value any limited series of tests that might be given. In the same way there is no evidence that copper segregates, experience pointing rather to perfect uniformity. A story has been the rounds of the trade journals of a copper wire which crystallized out in the head of a rail, but, unfortunately, no method is known by which the phenomenon can be duplicated, since such rails might be of great value in electrical work.

"Steel may contain up to one percent of copper without being seriously affected, but if at the same time the sulphur is high, say 0.08 to 0.10 percent, the cumulative effect is too great for molecular cohesion at high temperatures, and it cracks in rolling. This tearing occurs almost entirely in the first passes of the ingot, so that it is of little importance to the engineer who is concerned only with perfect finished material. In the purest of soft steels containing not more than 0.04 percent. of either phosphorus or sulphur, the influence of even 0.10 percent of copper may be detected in the less ready welding of seams during the process of rolling, but ordinarily when the sulphur is below 0.05 percent, the copper injures the rolling quality very little, even if present in the proportion of 0.75 percent. In all cases the cold properties seem to be entirely unaffected.

"These conclusions are not founded on any limited series of tests on special alloys; they are the fruit of years of experience in the making of millions of tons of cupriferous steels, and it is quite certain that any baneful influence of this constant companion would have been felt in the many investigations which have been made into the mechanical equation of structural metal."

A search of the literature substantiates Campbell's statement, for many of the older irons and steels contain appreciable quantities of copper. Unfortunately, few analysts determined the copper in iron or steel. Even then the copper content as reported was probably low, according to A. L. Colby, who discussed the

determination of copper in steel in an article in the *Iron Age* for November, 1899. He investigated the determination of copper in iron and steel and found that the early analyses, owing to faulty methods, did not show any or but little copper when there was considerable present.

While such cupriferous steel and wrought iron was made previous to 25 or 30 years ago, the character of the ore used for making Bessemer steel in this country has changed, so that at the present time little cupriferous steel is made by that process. This is confirmed by the results of the copper analyses made on a large number of fence wires. With one exception, all of this wire is of steel and probably Bessemer steel. Most of the older barbed wires contained copper in varying amounts, while the wire of recent manufacture contains but a trace, less than 0.02 percent. Two samples of wrought iron fence wire were analyzed. These two strands were made by the same firm and contained little copper.

Apparently the present-day wrought iron is likely to have copper in it. This has been demonstrated by several analyses of wrought irons of recent manufacture brought to the attention of the writer, and also by the report of Committee A-5 on Corrosion of Iron and Steel of the American Society for Testing Materials.[9] The Committee reports with reference to the analysis of the wrought iron sheets to be used in the extensive corrosion tests of roofing sheets:

> "It is interesting to note that the puddled irons have a high copper content, probably due to the use of eastern Pennsylvania ores, and an effort will be made to secure a genuine puddled iron substantially free from copper."

The present open-hearth steel contains varying amounts of copper, as shown in the woven wire fence analyses of Table I. Analyses of open-hearth steels show that the amount of copper present varies over a considerable range in successive heats, depending upon the nature of the scrap used. Where open-hearth steel is used for fence-wire this variation in the different heats will show up in a difference in the rate of corrosion of the different strands of wire. This is apparent in Table I, where the copper content of the horizontal strands of a woven wire fence made of basic open-hearth steel varied, the strand low in copper showing the greatest corrosion.

WHY ARE COPPER-STEELS A RECENT DEVELOPMENT?

The making of copper-steels is not a new art by any means. Irons and steels of ancient origin contain varying amounts of copper. The deliberate adding of copper to steel and iron to secure the necessary resistance to corrosion, however, is a new process which is now being used by several manufacturers. The manufacturers who make iron and steel products that are subject to corrosion are fortunate if they have available iron ores or pig iron containing sufficient copper, since copper is not eliminated in any of the refining processes.

Cushman, in Bulletin No. 5 of The Institute of Industrial Research,[8] after quoting H. H. Campbell on his experience in the making *"of millions of tons of cupriferous steels"* (see last paragraph of quotation from Campbell in this paper), states:

> "There would seem to be little use in pursuing this subject further; it is sufficiently well shown that copper-bearing steels are not new, and that those who make them in millions of tons had never claimed for them the valuable quality of extraordinary resistance to corrosion. If they really possessed this quality it is remarkable that the point should have escaped the notice of so many able contemporaneous metallurgists."

It *is* remarkable that so many metallurgists overlooked the fact that copper-steels showed high resistance to corrosion. But a closer study of the conditions under which these metallurgists were working readily explains this oversight.

Previous to 1860, wrought iron did not have its present competitor—mild steel. At about this time both the Bessemer and open-hearth processes were put in operation, and mild steel began to be a competitor of wrought iron. It was not long before statements appeared in the literature relative to the comparative rate of corrosion of wrought iron and steel. The makers of wrought iron were jealous of the steel makers and their attitude is reflected in the literature.

With the intense rivalry between the manufacturers of wrought iron and steel, it was natural that the metallurgists should confine their efforts to determine which one of the two was more resistant to corrosion. Few of the analyses available give the copper content of the wrought iron or steel of that time, but it is evident that copper was more often present than absent.

The attention of the metallurgists was turned toward the effect of copper on the physical properties of iron and steel because it was usually present. The idea was prevalent that copper made steel red-short, and ores containing copper were usually rejected in Sweden. As early as 1864 Percy's book on the Metallurgy of Iron and Steel devoted several pages to the effect of copper on the physical properties of iron and steel, and he quotes several experiments by various investigators to show that copper is not detrimental.

During the period from 1860 to 1880 the metallurgy of steel developed rapidly, and its chemistry was studied closely. Carbon, manganese, phosphorus, sulphur and silicon in various proportions gave steel widely different properties. Copper was sometimes absent, but more often present in small amounts, though seldom exceeding 0.20 to 0.30 percent, and investigation showed that it had practically no effect on the physical properties. Therefore, it was natural that the investigators should study the effect of carbon, manganese, phosphorus, sulphur and silicon—elements that were known to affect the physical qualities of the metal—with reference to their effect on corrosion, rather than copper, which apparently had practically no influence on the physical properties of the metal.

The general trend of the discussions along corrosion lines since 1880 is brought out in the following paragraphs.

W. E. Koch, while discussing the paper of A. Willis on "Reactions in the Open Hearth Process," before the British Iron and Steel Institute in 1880 (vol. 16, p. 120) states incidentally that he had been making a great many experiments in the corrosion of boiler plate and he thought it was much more due to the quantity of sulphur and phosphorus in the plates than anything else. A high manganese did not seem to affect the corrosion at all. He had had plates with 0.8 to 0.9 percent manganese and a low percentage of carbon which corroded less than when sulphur and phosphorus were in considerable quantities.

In a later communication to H. M. Howe,[10] Koch expressed the opinion that steel free from blowholes does not corrode easily, and that the function of the manganese is to free the steel from blowholes.

Another interesting point brought out in the discussion of Willis' paper was that copper was usually present in considerable amounts in manganese ores. The question was raised as to whether the presence of several percent of copper in the ferro-manganese added to the steel would injure its physical properties. The addition of manganese to the steels of that period evidently introduced appreciable quantities of copper. The effect of this copper on the corrosion of the steel was not touched upon.

W. Parker,[11] in 1881, reported an extensive series of experiments he had made on the relative corrodibility of wrought iron and steel. He found little or no difference between the two, but found that the more impure wrought irons resisted corrosion much more readily than those more highly purified. The latter contained much less cinder than the more impure material.

Parker's work started a large amount of discussion and some extensive work was started along the same lines by the Krupps, in Germany, to determine whether steel or wrought iron was more resistant to corrosion under various conditions. The work was carried out by H. Otto,[12] and covered a period of nearly four years—from 1882 to 1886—but the results were not published until 1896. The experiments were to determine what influence manganese, silicon and carbon had on the rate of corrosion, but no conclusions could be reached from the results. The interesting feature of the analyses of the various steels used show them to contain from 0.12 to 0.13 percent of copper, while the wrought iron contains from 0.05 to 0.06 percent. Otto does not discuss the influence of copper, though his analyses show its presence. The presence of the copper in the steel was probably the cause of its durability.

In 1895, Howe[10] discussed the relative corrodibility of wrought iron and steel. He makes the following statement, which is highly significant: "Under identical conditions of exposure not only different classes, but even apparently like pieces of wrought iron corrode at very unlike rates and so do apparently like pieces of steel. * * * It is generally believed that impure wrought irons as a class corrode much less than pure ones."

Up to 1900, none of the investigators had considered the effect of copper in the corrosion of wrought iron and steel. Copper was

not discussed when the corrosion problem was considered, although copper was known to be present in many irons and steels. The experiments were directed to determine the relative durability of wrought iron and steel and to determine the effect of carbon, manganese, phosphorus, sulphur, and silicon. While several investigators determined the effect of copper on the physical properties of iron and steel, they evidently were not interested in the corrosion problem.

Several had noticed at various times that the copper steels dissolved much more slowly in acid solutions, but did not proceed any further than this in their experiments.

Williams'[6] work in 1900 was the first to point out the beneficial effect of copper in steel and wrought iron. This was followed during the next year by the work of Stead and Wigham.[7]

Even after the convincing results of these investigators on the effect of copper in steel on corrosion, Cushman, in 1905, while studying the corrosion of wire fences, evidently failed to determine the copper in the fence wires, though it now appears that this is the most important element in iron and steel as affecting the corrosion of those materials. The importance of copper was not yet appreciated and the problem was attacked along the same lines that had been previously used, namely, the effect of the five elements, carbon, sulphur, silicon, phosphorus, and more particularly manganese. It was not until seven years later that the beneficial effect of copper was recognized by the iron and steel industry.

CORROSION UNDER TREES, BUSHES, GRASS, ETC.

The writer's observations on the corrosion of galvanized wire next to the ground, in bushes, etc., confirm in part the findings of Cushman. It was found that wherever galvanized wire was in contact or covered with weeds, brush, bushes, under trees, or adjacent to the points where stapled to trees or posts, the galvanizing was found to be much more durable than where it was in the open and subjected to the wind and sunshine. Some wires that evidently had been in service from 20 to 25 years were found with the galvanizing in good shape. Even where sulphur dioxide fumes caused rapid corrosion, this protective effect was observed.

The galvanizing, when found intact under such conditions, was

usually coated with a hard white film, probably a basic carbonate of zinc. The effect of this salt as a protective agent, and the conditions under which it forms, have not been studied, but it may have an important bearing on the corrosion of zinc.

The effect of sunlight also may be a factor. This is suggested by Friend's[13] work, in which he finds that sunlight accelerates the rusting of iron in the presence of water.

While the galvanizing under such unfavorable conditions is much more durable than under apparently favorable conditions, it does not appear that iron or steel shows the same characteristics. Many observations have been made on woven wire fences that have been erected for such a period of time that the galvanizing has been entirely removed. The bottom portion of the fence which has been in contact with the wet grass and weeds has been badly corroded, while the upper portion is in much better condition. It was observed that the steel in contact with the grass was covered with a loose, yellow flaky rust, while that further up was covered with a deep-red adherent rust.

These observations cannot be considered conclusive and more work on this point is necessary. In many places the burning of the dry grass has caused conditions which make any observations worthless.

If it can be definitely proved that iron and steel corrode at an accelerated rate at the bottom of a fence or in brushes and weeds, and that the galvanizing is much more durable under those conditions, Cushman's hypothesis, that the corrosive electrolytic action is diminished by keeping the wires electrically neutral through frequent connections to the earth, is untenable.

SUMMARY.

1. It has been definitely established that the durability of old steel fence wire is due to the presence of copper.

2. Manganese does not increase the corrosion of steel, and its absence does not decrease the corrosion.

3. The copper in the early steel and wrought irons came from the copper-bearing ores of the eastern United States and imported ores. The proportion of copper-steel made decreased with the increasing importance of the copper-free Lake Superior ores.

4. Steel fence-wire containing copper is as durable as wrought iron.

5. Present-day steel fence wire usually does not contain copper, and therefore corrodes rapidly.

6. Under ordinary atmospheric conditions zinc corrodes more slowly under conditions favoring rapid corrosion of iron, and *vice versa,* iron corrodes more slowly under conditions favoring rapid corrosion of zinc.

7. The life of fence wire is dependent upon the quality of both the galvanizing and iron or steel base. Since the galvanizing is usually thin, the life of the fence depends principally upon the iron or steel base, which should be highly resistant to corrosion.

The writer wishes to thank the Mineral Point Zinc Company for generously allowing him to publish data which were obtained for them in determining the damage done to fence wire by sulphur oxide fumes in the vicinity of Mineral Point, Wisconsin.

Since the completion of this investigation by the writer, a much more complete investigation of the fence-wire corrosion problem involving the complete analyses of a much larger number of barbed wires is being jointly made by Mr. D. M. Buck, Metallurgical Engineer of the American Sheet and Tin Plate Company, and the writer. The results, confirming the work of this paper, will be published at an early date.

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C. F. Burgess Laboratories,
Madison, Wis.
August 30, 1917.

DISCUSSION.

FRANK N. SPELLER[1]: Ten years ago a research of this character would have had a much broader title. I congratulate the author on confining the title strictly to the limits of the class of material tested. We have not yet learned all that we would like to know about the mechanism of corrosion, but it has been well established beyond question that conditions of service have much more to do with corrosion than the material itself, and that it is very unsafe to generalize from any particular experience under one set of conditions. This is mentioned merely in a cautionary manner, for I expect that when this paper gets into commercial circles the excellent showing which copper-steel has made under atmospheric conditions will lead some who are not well informed to hail this material as another panacea for all corrosion troubles, just as was the case with ingot iron and wrought iron not very long ago.

In the case of pipe carrying water, for example, we have tried out copper-steel and can confirm everything the author says in regard to that material so far as the outside of the pipe is concerned when exposed to atmospheric conditions, but on the inside the water seems to attack copper-steel as rapidly as ordinary steel without copper. Many tests have been made in service in

[1] Metallurgical Engineer, National Tube Co., Pittsburgh, Pa.

the same lines carrying ordinary steel pipe, copper-steel and wrought iron, all of which show practically the same results after two or three years' service with respect to the depth of the pitting. While the conditions are nominally the same, that is, the presence of water and air, in one case, on the inside of pipe, we have a relatively large proportion of water. On the outside, on the other hand, we have the reverse, *viz.*, a large proportion of oxygen and a very small amount of water. The oxygen in each case is the depolarizing agent, and the proportion present seems to govern the relative rates of corrosion of these materials.

JAMES ASTON[2]: I wish to take the same stand as Mr. Speller, and interject a word of caution in interpreting this paper.

Aside from the interesting and valuable data which Mr. Storey puts forth, the paper itself points to the complexity of the corrosion problem, because of the many factors involved, and it also points to the strong moral: Don't jump at conclusions. I do not know how many theories have been expounded and false conclusions drawn from misinterpretation of data, due to the fact that all of the factors have not been taken into account.

These statements are intended as general remarks, and have no application whatever to Mr. Storey's conclusions.

Now, aside from the protective coatings, the trend in the production of materials to withstand corrosion has in late years come down to three different types. The oldest has been wrought iron. For a long time, even today among people who have not made a study of the problem, wrought iron is assumed to owe its stability to the purity of its base. My own work, which now deals largely with that material, has forced me to the belief that while the purity of the base material is a considerable factor, it is after all the secondary factor, especially in view of the established fact that rust once formed is an important element in its progression.

Wrought iron owes its resistance to the physical factor; that is, to the multitude of slag barriers which are an integral part of well-made puddled iron.

The second and later trend in the development of materials was the pure iron made by open-hearth methods, the slagless metal with a pure base. While this is a big step forward, it after all

[2] Metallurgical Engineer, A. M. Byers Co., Pittsburgh, Pa.

touched only on the secondary factor, and therefore did not accomplish the desired end.

The most recent development is by using alloying materials.

Unfortunately, through force of circumstances, Mr. Storey's paper does not tell us the comparative value of pure iron. It does tell us, however, that both wrought iron and copper-bearing materials have fulfilled their function; that is, they have a good measure of stability. In this connection I wish to take exception to one of Mr. Storey's statements, where he said that without exception those wires which withstood corrosion were copper-bearing; and those which did not, were not copper-bearing. He cites only a few cases where wrought iron was found, but it invariably resisted corrosion and was free from copper.

Mr. Storey deals with fence wire. That is, he deals with a special commodity under a special condition of service. If we accept the electrolytic theory of corrosion, we are concerned with not only the electrodes, which is the material itself, but also with the electrolyte, consisting of moisture, and the depolarizer which is the oxygen of the atmosphere. The last two factors may become of even greater importance than the electrodes, the material itself.

Take a general condition: Materials exposed to the atmosphere have an abundance of oxygen and a paucity of the electrolyte. On the other hand, pipes and tubes usually have an abundance of electrolyte and a relative scarcity of depolarizer. So we must be very cautious, considering that material and two other factors are involved, we must be cautious in drawing conclusions not to say that the conditions apply to other conditions of service. And we cannot say then that any one of these three methods for the production of corrosion-resistant materials is necessarily the right means to every end.

D. M. Buck[3]: I am quite sure that Mr. Storey did not intend to ask you gentlemen to believe that copper-steel is a panacea for all ills. He has collected a large amount of very valuable data, which proves the remarkable influence of a small copper content on the corrosion rate of fence wire. Mr. Storey wrote me several weeks ago telling of the information he had obtained, and

[3] Metallurgical Engineer, Am. Sheet & Tin Plate Co., Pittsburgh, Pa.

since these results corroborated work which I have done during the past seven or eight years, I went out to Wisconsin and collected a large number of samples of fence wire from the locality mentioned in Mr. Storey's paper. I have analyzed these wires complete, including copper, and I have with me thirty or forty such samples to which I have attached tags, showing the complete analysis.

I am entirely in accord with Mr. Storey's conclusions, since my own work had demonstrated to me that, contrary to opinions expressed by Dr. Cushman in his pamphlet on fence wire, manganese does not harmfully influence steel from a corrosion standpoint. I am also firmly convinced of the beneficial effects of the introduction of a small percentage of copper.

Mr. Storey has mentioned the influence of the rust film on the corroded wire. The peculiarity about copper-steel is that, after approximately 12 months' exposure, rusting action, due to the formation of the protective film, nearly if not completely stops. I have demonstrated this fact by observations on many full-size corrugated sheets, and also by weight, by exposing large numbers of small test pieces and taking down and reweighing at regular intervals a certain number of each grade. Normal steel continues to lose weight until complete destruction, the corrosion rate rather increasing toward the end of the period; whereas in the case of copper-steels after ten or twelve months' exposure, corrosion practically ceases, a dense almost vitreous coating being formed on the surface.

PRESIDENT C. G. FINK[4]: Mr. Buck submitted to us some time ago copper-steel sheets. These were put through various tests and compared with other steel sheets. Mr. Buck's withstood all conditions except those which prevail on the Atlantic and Pacific coasts. Exposed to a seacoast atmosphere, copper-steel did not stand up. It would be wrong, however, to conclude that copper-steel was "no good" because it did not stand up under these severe conditions. I am interested in hearing whether any experiments have been made along the line of getting a copper-steel which will withstand not only inland weather conditions but seacoast conditions as well, especially where considerable amounts of sodium chloride deposit on the steel.

[4] Director of Research, Chile Exploration Co., New York City.

D. M. Buck: Answering Mr. Fink's question, I have made corrosion experiments in the salt air of the Atlantic Coast, and also by complete immersions in salt brine. In the latter case I find that all materials show a rather rapid corrosion rate, and that the differences are not as great as in atmospheric conditions.

However, from a series of tests including several hundred sheets of various grades of material, exposed to the atmosphere of Atlantic City, I find approximately the same advantage for copper-steel over other materials as has been found in other districts. General corrosion on all grades is greater in salt air than in pure air and less than in the atmosphere of a mill district.

President C. G. Fink: My tests on the effect of sea atmosphere were more severe than yours, because I kept bright copper sheet in contact with the steel sheet.

J. W. Richards[5]: I have not the results of any experiments to record, but I wish to call attention to the fact that the barbed wire shown offers favorable conditions for local electrolysis, inasmuch as the two wires are twisted together and would therefore hold moisture between them, and so give the best conditions for electrolytic corrosion. If there is the slightest chemical or physical difference between the strands which are twisted together, the moisture between gives maximum conditions for corrosion taking place. A straight wire, untwisted, would probably not corrode at the same rate as these twisted wires, and the results could not be transferred to the corrosion of sheet. If my remarks are correct, the results apply only to the twisted wire, and not to straight wire or sheet.

O. W. Storey (*Communicated*): The misinterpretation of data, which often were incomplete, and the application of the results of a single series of corrosion tests when made under a single set of conditions to the entire range of conditions has been the cause of many misunderstandings and bitter controversies during the past ten years. With this in mind, the conclusions reached in this paper were confined to the corrosion of fence wire and similar materials under atmospheric conditions.

I do not find any statement in my paper such as that to which Mr. Aston objects. On the contrary, I have been careful to point

[5] Professor of Metallurgy, Lehigh University.

out that without exception those *steel* wires which withstood corrosion were copper-bearing; and I also called attention to the fact that the wrought-iron wires, though practically free from copper, were also highly resistant to corrosion.

The work of Mr. Buck shows the importance of the protective rust-film on copper-bearing steels. Mr. Buck has kindly shown me some of these corrugated sheets which he mentions. The rust on the copper-bearing steel was hard and vitreous like a hard baked enamel, while that on the ordinary steel was flaky and easily removed.

Professor Richards states that the results of this paper should be applied only to twisted wires because of the electrolytic corrosion which he believes takes place. This objection is fully answered in the paragraph at the bottom of page 213.

The President states that in his corrosion tests of copper-steel in a sea atmosphere, he kept a bright copper sheet in contact with the steel sheet. This condition, of course, results in an accelerated test that cannot be compared to the ordinary corrosion tests.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President C. G. Fink in the Chair.

ELECTROLYTIC ZINC EXTRACTION AT TRAIL, B. C.

By E. H. Hamilton.*

[Abstract.]

The composition of the ore treated is given, the crushing analysis, details of the roasting operation, including temperatures on the various hearths, the leaching in "Pachucas," neutral classifiers and Dorr thickeners, purification of electrolyte before electrolysis, construction of electrolytic tanks, details of the electrolysis, and analysis of the zinc produced.

At the Trail, B. C., plant of the Canadian Mining & Smelting Co., the zinc plant started operating in April, 1916. The ore on which the plant principally depends comes from the Sullivan mine and the portion assigned to the zinc plant has an approximate analysis of:

Lead	14	percent
Iron	23 to 31	"
Insoluble	4	"
Alumina	3	"
Lime	2	"
Magnesia	2	"
Sulphur	24 to 29	"
Zinc	19 to 24	"
Cadmium	0.04	"

(1) The ore is crushed in tube mills to about the following size:

+ 48	3	percent
+100	10	"
+150	3	"
+200	12	"
—200	72	"
	100	"

(2) The ore is then roasted in Wedge roasters having 7 floors and diameter of 26 ft. (8 m.), with two arms to the floor and re-

* Manager, Consolidated Smelting & Refining Co. of Canada, Ltd., Trail, B. C.

volving once in 4 minutes. Forty tons are roasted per day from 25 percent down to 5 percent sulphur. The temperature of the hearths is approximately:

1st floor	...	
2nd "	950°	F. (510° C.)
3rd "	1,000°	F. (538° C.)
4th "	1,100°	F. (593° C.)
5th "	1,100°	F. (593° C.)
6th "	1,050°	F. (566° C.)
7th "	...	

(3) The calcine is then treated with partly neutralized spent electrolyte in a series of "Neutral Pachucas" (or Brown tanks) until the solution is quite neutral. The overflow goes to Dorr classifiers.

(4) The sand (mostly over 100 mesh) is removed in Dorr classifiers.

(5) The neutralized overflow from the "Neutral Classifiers" is then transferred to a series of "Neutral Dorr Thickeners," where it is treated by counter-current system. (For overflow see 7.) The last underflow from the "Neutral Thickeners" is then treated with the sands in a series of "Acid Pachucas," which deliver overflow to acid thickeners, the pulp running counter-current to spent electrolyte from the tank room.

(6) The underflow from the last acid thickener is washed with water and is then pumped to the slime pond. The last overflow is delivered to the series of "Neutral Pachucas" (see 3, above).

(7) The overflow from the "Neutral Dorr Thickeners" is transferred to and passed through a series of three "Precipitating Pachuca" tanks, where it is agitated with atomized zinc to precipitate the copper and cadmium.

(8) The crude electrolyte is then filtered and delivered to tanks which supply the electrolytic tank room.

The electrolytic tanks are made of concrete, lined with asphalt, and are in cascades of eight tanks in series. Each tank is 82 in. (2.14 m.) long by 27 in. (90 cm.) wide and 42 in. (107 cm.) deep. Each tank has 17 lead anodes and 16 aluminum cathodes, the cathodes have an area of 10 sq. ft. (0.9 sq. m.) and the anodes 9 sq. ft. (0.8 sq. m.). The current density is 24 A. per sq. ft. (265 per sq. m.), the voltage across the tanks is about 3¼, the

electrolyte contains about 7.5 percent zinc, 0.014 percent iron, 0.003 to 0.008 percent cadmium, 0.0003 percent copper, and sometimes manganese, lime, magnesia, and other impurities coming into the solution from the ore. Everything in the electrolyte comes from the ore. The sulphuric acid is made in the plant from ore from the Sullivan mine. The average spent electrolyte from the electrolytic tanks is about 3 to 5 percent zinc, 0.013 percent iron, 5 to 7 percent free sulphuric acid.

The temperature of the solution in the tanks varies from 30 to 45° C.

The cathode zinc is stripped from the aluminum cathodes and is melted in a reverberatory furnace. The production is about 50 tons per day. The bars weigh 50 lb. (23 kg.), and have an analysis of about—

Zinc	99.92	percent
Lead	0.03	"
Cadmium	0.040	"
Iron	0.007	"
Copper	0.002	"

The whole system is continuous, and in the leaching department by continuous counter-current.

Trail, B. C.,
Sept. 24, 1917.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President C. G. Fink in the Chair.

THE FUTURE OF ELECTROLYTIC ZINC

By Thomas French.*

[Abstract.]

A discussion of the comparative merits of the retort process and the electrolytic process of extracting zinc from its ores, particularly considering the cases of concentrates and of lead-bearing zinc ores. The conditions for satisfactory roasting preliminary to leaching are considered, the leaching process, and the conditions for depositing pure zinc, as determined by the purity of the electrolyte. Detailed costs of the electrolytic process are given, as compared with the retort process treating the same ore, showing that the former method has a good prospect of ultimately superseding the latter.

In the last two years a large quantity of high-grade electrolytic zinc has been produced in the United States and Canada, and at the present time many other plants are projected. In view of this rapid progress the question has often been asked: "Will the older retort process of smelting be superseded by the electrolytic deposition of zinc?"

The reply to this question is generally given in an unqualified negative, apparently justified by the rather unsatisfactory recoveries of zinc obtained by those who have adopted the electrolytic process. The poor recovery is attributed mainly to the difficulty of roasting the ore without the formation of ferrate of zinc, which is very difficult to dissolve in the acid effluent from the electrolytic vats, but also to the difficulty in dealing with the filtration of slimy acid and neutral solutions.

* Manager, French Complex Ore Reduction Co., Nelson, B. C., Canada. (Manuscript received Sept. 22, 1917.)

We hear of great variations in the amount of zinc that can be dissolved from the roasted ore. Sometimes a 90 percent extraction of the zinc in the ore is obtained, and at other times only half, or even less than that amount, from the same ore. The average recovery of zinc appears to be less than 70 percent by most of those engaged in the manufacture of electrolytic zinc from ores or concentrates containing about 40 percent of zinc.

If an extraction of 90 percent of the zinc can at times be obtained, and at other times can not be obtained from the same ore, then assuredly, sooner or later, the reason for the disparity will be found. That being so, and taking into consideration several weighty factors favoring the electrolytic process for the reduction of zinc from its ores, there is more than a strong probability that the retort process for the reduction of zinc is doomed in the near future.

In forming this opinion, one of the chief considerations is: That electrolytic zinc is being produced either by lead or copper manufacturers, and may be considered to be a by-product. It is true that some of these manufacturers are treating custom ores, but this is only to take advantage of the very profitable price of zinc which has ruled for some time, and to fulfil large contracts. The primary reason for the erection of these electrolytic plants was to extract from their own ores the zinc which had previously either been wasted or not worked at all.

In the case of the lead smelter, especially those whose ores contain much zinc, it is imperative that the zinc should be extracted, in order that the lead may be profitably smelted. Before long a modern lead smeltery will not be considered complete without the adjunct of an electrolytic zinc-reduction plant, if it is within reach of electric power at a reasonable rate. The competition of this by-product zinc will itself seriously affect the output of the retort smelters.

With proper adjustment of the roasting furnace conditions, it it not a difficult matter to obtain extractions of 90 percent of zinc from ores containing 15 to 25 percent zinc, and in the latter case with as much as 25 percent of iron. As far as the author's experience goes, furnaces of the Wedge and Herreshoff type are

admirably adapted to the roasting of this class of ore. A large Wedge furnace, with 7 hearths and a diameter of 22 ft. 6 in. (6.8 m.) is capable of roasting about 1¼ tons of this ore per hour, with unskilled labor, and gives a more satisfactory product than the smaller hand-rabbled furnaces. After roasting zinc concentrates containing 45 percent of zinc there is little difficulty in extracting as much as 95 to 97 percent of the zinc.

The successful electrolysis of zinc from sulphate solutions has not been an easy matter, and it is only within recent years that the difficult problem of depositing high-grade zinc has been satisfactorily accomplished on a commercial scale. The principal requisite is that the electrolyte shall be quite free from certain impurities. The methods by which freedom from these impurities is assured are now very well understood, and with experienced superintendents there is little difficulty in obtaining a high efficiency in that part of the process.

When certain underlying principles are recognized, the difficulty encountered in dealing with the filtration of the acid and slimy solutions also largely disappears, although it has been a very serious one to the uninitiated. In the dissolving of the zinc from the roasted ore there is nothing which any competent chemical engineer can not undertake, and any other operations connected with the electrolytic process are either of little or no difficulty, or are common to the retort process.

In comparing the two processes, I do not think there is any more experience required in the electrolytic process than in retort smelting, and I shall not be surprised if within the next year or two the electrolytic is preferred as the simpler of the two. It is certain that, with the exception of the care of the electrical machinery, the commonest unskilled labor only is required to produce electrolytic zinc, whereas the retort process requires not only many physically strong, but also very skilled and highly paid men. The amount of labor necessary in the electrolytic process is, however, greater than that required in retort smelting, and also, as a general rule, the current to produce a ton of zinc costs much more than the gas or fuel to produce a ton of spelter. The electrolytic is as much dependent on cheap power as the retort

process is on cheap fuel. The operation of roasting in Wedge furnaces is, however, less costly than that in the special furnaces used for roasting the ore for retorting, in which nearly all the sulphur must be eliminated. The labor and power costs are in favor of the retort process.

The following costs for the treatment of run of mine ore of a complex nature are based on zinc at 5 cents per lb. St. Louis; lead at 5 cents per lb.; and silver at 60 cents per oz. Eighty-eight percent recovery of zinc is assumed to be made. A typical composition of such an ore may be taken as zinc 30 percent, lead 15 percent, and silver 12 oz. per ton. The average amount of current required to deposit a ton of zinc is less than 3,000 K.W.H., measured at the vats, when the process is well conducted and the solutions are pure and in good condition. To this must be added the ordinary motor, generator and other losses, and also the current incidental to the operating of the factory. Four thousand K.W.H. have therefore been allowed per ton of zinc produced, although this amount is more than is necessary in practice.

Ninety-five tons of ore produce 25 tons of zinc.

		Per Ton Zinc	Per Lb. Zinc
100,000 K.W.H. @ ¼ cent	$250.00	$10.00	0.50 cent
Labor @ $3.00, and management	310.00	12.40	0.62 "
Fuel and supplies	60.00	2.40	0.12 "
Roasting, including labor and fuel	120.00	4.80	0.24 "
Insurance, maintenance and depreciation	250.00	10.00	0.50 "
	$990.00	$39.60	1.98 "

Freight on the zinc naturally depends on the location of the electrolytic plant, and as this may be situated in a position difficult of access, we shall allow the ample average sum of $15.00 per ton of zinc:

		Per Ton Zinc	Per Lb. Zinc
Costs as above	$ 990.00	$ 39.60	1.98 cent
Freight on zinc	375.00	15.00	0.75 "
	$1,365.00	$ 54.60	2.73 "
Balance, cost of ore and profit on zinc alone	1,135.00	45.40	2.27 "
25 tons zinc @ 5 cents	$2,500.00	$100.00	5.00 "

In the case of an ore with the same content of lead and silver, but with 25 percent zinc, the cost of production would be increased to the extent of 0.05 cent per lb. of zinc. Complex ores of this composition are very plentiful, and are of no value unless treated in conjunction with the electrolytic process, for it is impossible to recover the lead and silver by smelting unless the zinc is partially removed. After the sulphur has been nearly eliminated by roasting, and the zinc has been extracted, the residue has only about one-half the original weight, and the percentage content of lead and silver is doubled. The zinc that has been recovered not only pays for the cost of the ore but leaves a considerable profit, and the lead smelter has now a desirable smelting product, containing nearly 30 percent lead and 24 oz. of silver per ton, which costs him nothing, as its cost has been charged to the zinc.

Smelting 48 tons residue, containing 30 percent lead and 1,140 oz. silver, @ $5.00	$ 240.00	
Freight on 13 tons lead and silver @ $10.00	130.00	
		$ 370.00
12.65 tons lead @ 5 cents	$1,265.00	
1,083 oz. silver @ 60 cents	649.80	
		$1,914.80
Deduct smelting and freight		370.00
Profit on lead and silver		$1,544.80

No allowance is made for the recovery of vitriol, as it is common to both processes.

Just as soon as the lead or copper smelter recognizes that a good extraction of zinc can be made from his zinc-lead or copper ores, we may expect him to erect an electrolytic zinc plant as an adjunct to his smelter, even though the price of zinc should fall so low that he can make no direct profit from the zinc, provided, of course, that he is favorably situated with regard to cheap power.

There is another aspect of the question that has not yet received sufficient attention, which will have the effect of turning the balance in favor of the electrolytic process, should it be found that the retort process is still able to hold its own. In the electrolytic process the presence of manganese, in at least a small quantity,

is an essential. Its principal function is to prevent sulphating or destruction of the anodes in the electrolytic vats, but it has also other valuable properties, chiefly in purifying the electrolyte. A large part of the manganese is deposited at the anode as hydrated manganese dioxide, concurrently with the deposition of zinc on the cathode. When the zinc ore contains much manganese, which is very frequently the case, the manganese dioxide recovered forms a very valuable by-product. No extra current, or rather, as far as the author's measurements have gone, only a negligible extra quantity of current is required to deposit the manganese. Unfortunately, the particular kind of roasting which is necessary to make the zinc soluble is not that which is most suited to the best recovery of manganese, and therefore, when manganese is present in a zinc ore only part of it becomes soluble. There is no reason, however, why low-grade manganese ores should not be treated in a special furnace, and the solution obtained by leaching added to the electrolyte, and recovered as manganese dioxide in the electrolytic vats. The manganese dioxide is particularly well suited for use in dry batteries, quite apart from the demand that exists for manganese in the steel industry. A small quantity of the manganese dioxide deposited is used for the regeneration of manganese sulphate or sulphuric acid. The solution of the zinc in the roasted ore may be conducted in such a way that there is very little or no loss of acid through the precipitation of basic sulphate of iron, but generally the iron and lead deplete the solution of more sulphate than the sulphate of zinc (of which there is usually a small quantity in the roasted ore) furnishes. Sulphurous acid from the roaster is brought into contact with the manganese dioxide, and manganese sulphate is formed, from which dioxide is deposited in the electrolytic vats, and the sulphuric acid with which it was combined is in this way added to the circuit.

There is still further evidence that may be adduced in favor of the future supremacy of the electrolytic process. It is well known that with 5-cent spelter in pre-war times, it was impossible for all except the most favorably situated retort smelters to make an adequate profit. The sulphuric acid recovered by some of the

smelters enabled them to tide over the bad times, until the surplus stock of spelter had been disposed of. At that time it was very difficult to obtain zinc concentrates, for at the price of $40.00 per ton of 60 percent concentrates, the miner could not make sufficient profit. So many mines were shut down at that time that various writers attributed the cause to actual depletion of ore in the mines. It has since that time been proved that the real reason was that it was unprofitable to work at the prevailing prices. In Canada, and especially in British Columbia, there are vast quantities of complex ores and silver-zinc-lead ores, which it has not been possible hitherto to concentrate to much above 45 percent. The retort process in Canada is impossible on account of the small supply of ore of sufficiently high grade. The concentrates are very impure, and therefore do not command a high price. These concentrates are, however, very easily treated by the electrolytic process. It is imperative that the British Columbia miner shall concentrate his ore in order to separate the valuable silver-lead contents; the zinc concentrates are merely a by-product. The latter generally carry a considerable quantity of silver, of which 65 to 70 percent is paid for by the United States smelters, after first deducting 6 oz. The price paid for the zinc is generally based on 5-cent zinc, $17.00 to $18.00 per ton being given for 40 percent concentrates. After deducting duty, penalties and freight to the nearest United States zinc smelter, the Kootenay miner receives only $5.00 to $7.00 per ton for his zinc concentrates. An electrolytic plant using these concentrates will prove to be a very serious competitor to the zinc smelter who can treat only high-grade concentrates. Even though he were able to treat 40 percent concentrates as profitably as 60 percent, the Missouri miner could not supply his 40 percent concentrates to the smelter at much under $27.00 per ton. The electrolytic plant would thus have an advantage of about $20.00 per ton in the cost of ore, which much more than compensates for the higher cost of production in that process.

It must not be forgotten that when zinc-lead ore occurs in veins, it must be concentrated in order to separate the more valuable lead contents, whether the miner is able to dispose of his zinc

concentrates or not. Many of them actually run the zinc down the creeks because there is no market for it.

There are, of course, the considerations of cheap electric power, the improvement in flotation methods of concentration, distance from markets, and many others, which affect the future of these rival processes, but I think it must be conceded that it is at least very doubtful whether or not the retort process will be able to survive the competition of the electrolytic process within a few years.

DISCUSSION.

MR. ————[1]: The author very carefully avoids stating the particular impurities which cause trouble in the deposition of zinc. This factor has a very large bearing upon the type of ore suitable for electrolytic treatment. The more electro-negative elements like copper, iron and nickel cause serious disturbances on the cathode plate if there is the least bad contact. Copper is the worst offender, but both it and iron are easily removed from solution, whereas the removal of nickel is a costly complex chemical engineering problem. For this reason ores containing nickel even to a small fraction of one percent will probably be unavailable for electrolytic treatment, at least with our present knowledge.

The estimate given for power is very conservative. If the process is to be used in places of higher power cost than that figured by the author ($21.90/K. W. year) a lower power consumption than 4,000 K. W. hours per ton must be regularly made. Thirty-five hundred K. W. hours per ton at the 2,200 v. primary should be a maximum. In the upper section of the cascade 100 percent current efficiency is easily made in running, while an average of 92 percent over 12-16 tanks is easily possible.

[1] Name of discusser could not be ascertained.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, Oct. 5, 1917, President Fink in the Chair.

AN IMPROVED ELECTRO-ANALYSIS APPARATUS

By J. L. Jones.*

[Abstract.]

Description of a six- and a ten-unit electrically-stirred electrolytic apparatus for quantitative chemical determinations, particularly for use on analysis of non-ferrous alloys. The electrodes and beakers are fixed; stirring is done by revolving tungsten rods. Each analysis receives its own current, graded by a rheostat from 0.5 to 6 amperes. A switch and push-button at each beaker enable the amperage and voltage to be determined at any time.

In the chemical laboratories of many of the larger manufacturing establishments, the volume of non-ferrous work is so great that the types of electro-analysis apparatus, supplied by the dealers in chemical supplies, do not prove adequate.

The apparatus which forms the subject of this paper has been in use in the laboratory of the Westinghouse Electric and Manufacturing Company for about two years. There were incorporated in its design the good points of the apparatus previously in use in our laboratory as well as those forms on the market which we had purchased and tried out from time to time.

In its present form the apparatus is strongly constructed, compact, easy of adjustment and current control, and capable of turning out a large amount of work in a minimum time.

We have built, and have in use, one ten-unit and two six-unit machines. Fig. 1 gives a view of the six-unit machine. One of the latter machines has been mounted on a convenient table and a demonstration will be made at this time of its operation.

* Metallurgist, Westinghouse Electric & Manufacturing Co., East Pittsburgh, Pa.

Fig. 2 gives the more important details of mechanism. They are:

A—Drive Shaft.
B—Leather Friction Driver.
C—Disc.
D—Thrust Ball Bearing.
E—Pressure Spring.
F—Pressure Screw.
G—Lock Nut.
H—Bearing.
I—Shaft.
J—Oil Hole
K—Fiber Bushing.
L—Large Electrode.
M—Small Electrode.
N—Stirring Rod.
P—Hard Rubber Plate.
R—Terminal Screw.
S—Hard Rubber Cup.

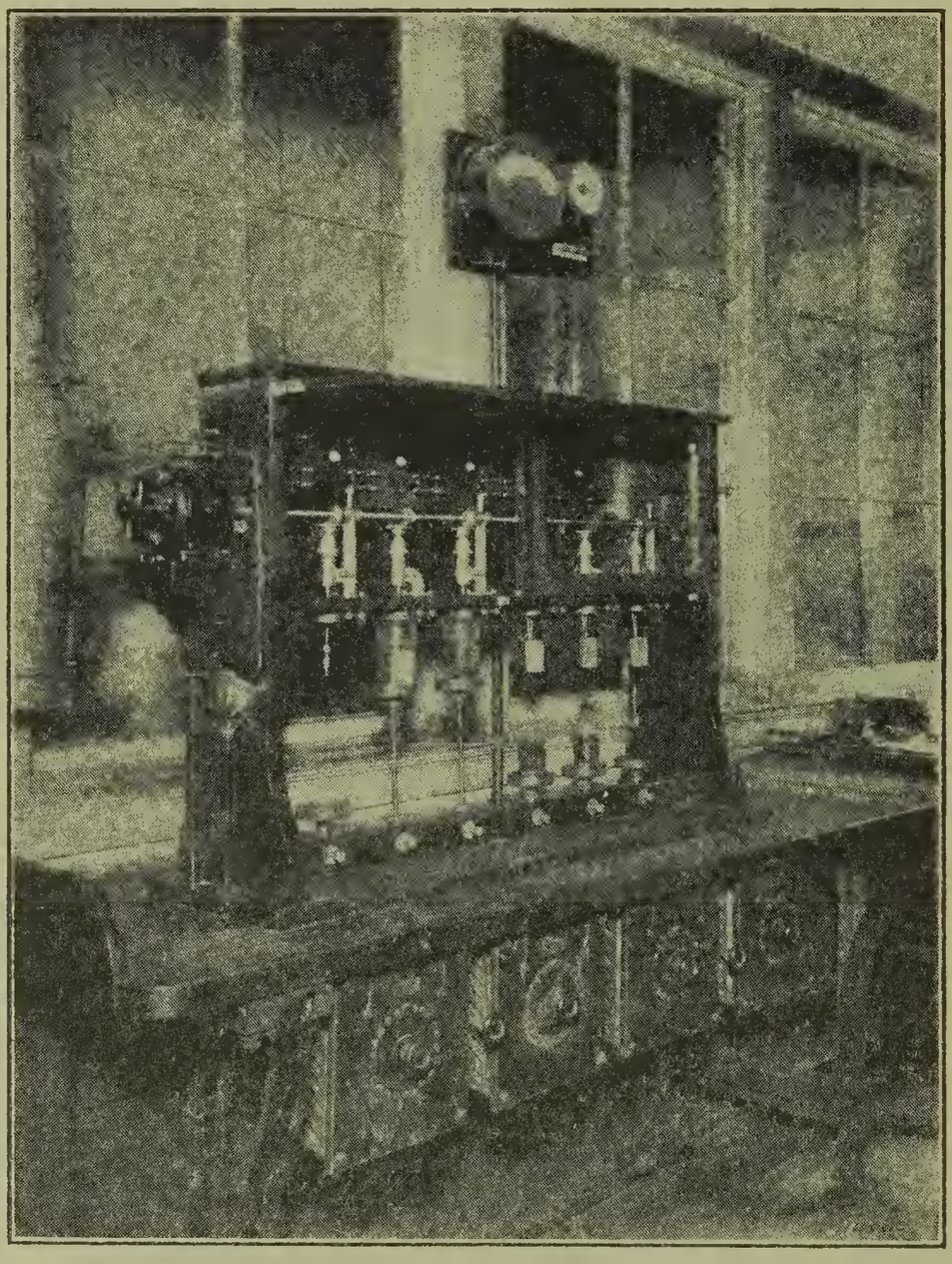

Fig. 1.

Drive Shaft, Etc.: The drive shaft is made of ground tool steel and is directly connected to a 1/12 H. P. motor, 1,140 R.P.M. The leather friction driver, disc and related parts furnish a means of throwing any spindle out of action at will and of also

modifying the speed of rotation. The pressure spring enables the leather friction driver to be fastened securely in any desired position on the disc and is a valuable accessory for this reason.

The leather friction driver and the thrust ball bearings give almost noiseless operation at this point.

Electrodes: The electrodes are cylinders of platinum made from 40-mesh wire gauze. The large electrode weighs about 30

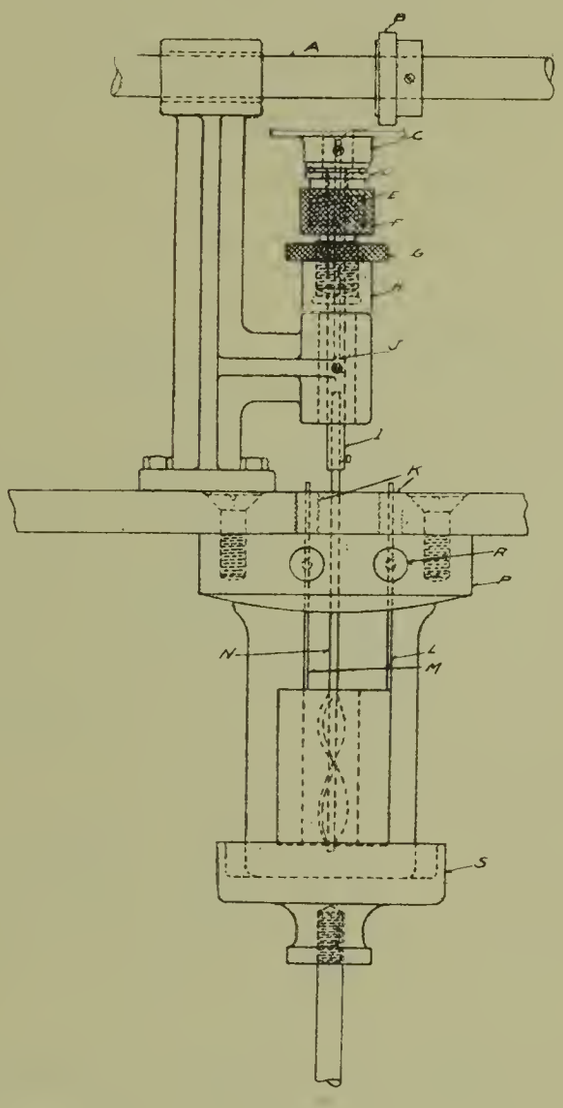

FIG. 2.

grams and the small electrode 16 grams; in other words, they are as light as is consistent with the work they have to do. They are stationary, and this enables them to be used with success for lead determinations, as the deposit adheres well, which is not the case when a revolving anode is used. The use of the gauze electrodes assures rapid deposition, and surface bubbles are to a large extent prevented. Although both electrodes are stationary and not subjected to the severe stresses due to rapid rotation,

considerable trouble has been had with them due to the supporting rods breaking. The platinum obtainable during the past two or three years has been very inferior in quality and the prices high. The manufacturer insists that his refining methods have never been as good as at present and that his platinum was never before as pure, but the facts are that electrodes bought five or six years ago and that have seen hard and continuous service are still in good shape, while the platinum in recently purchased electrodes is hard and brittle, the supporting rods frequently breaking and in some cases after only a few weeks of service.

Stirrers: Our stirring rods were at first made of platinum and were shaped like propellers. Their frequent breaking and the fact that it would have been necessary to make them very heavy to have enabled them to stand up led to the development of a new type of stirrer, which consists of a tungsten rod 3/32 inch (2.4 mm.) in diameter and 8 inches (20 cm.) long, with a glass screw fused upon it. These stirrers are stiff and strong, and have been found to be much cheaper and more satisfactory than those made from platinum. They can only be used in acid electrolytes, however, as in alkaline solutions the tungsten is attacked. Where it is necessary to work with alkaline solutions, as in the case of electrolyzing ammoniacal solutions of nickel sulphate, etc., a coating of glass may be spun over the entire surface of the stirrer that is exposed to the solution.

The speed of rotation of the stirrers may be varied from 300 to 1,000 revolutions per minute. To insure smooth running, the tungsten rods must be absolutely straight, and this requires careful selection. A set-screw has been found to be the most satisfactory method of holding the stirrers in position.

Hard-Rubber Plate: A hard-rubber disc or plate with a convex lower surface carries the terminal screws for the electrodes and fits closely over the beaker that holds the electrolyte, thus preventing loss of the liquid by projection or from the fine spray that occurs during the electrolysis. The holes in the disc for the supporting rods of the electrodes and for the stirrer are made as small as possible for similar reasons.

Hard-Rubber Cup: A hard-rubber cup or holder which can be adjusted to any desired height carries the tall glass beakers that

are used for the electro-analysis. Beakers of from 200 to 400 c.c. capacity are used, the larger sizes being necessary for large samples of copper that are being analyzed for impurities which are present in relatively small amounts. Where it is desired to heat the solutions, a small disc electric heater is placed under the beaker.

Instruments: The ammeter has a range from 0 to 10 amperes and by means of a shunt will indicate from 0 to 1 ampere. It is connected in such a way that readings may be taken at any spindle by throwing in the switch above the spindle. Corresponding voltmeter readings may be taken by means of the push-buttons.

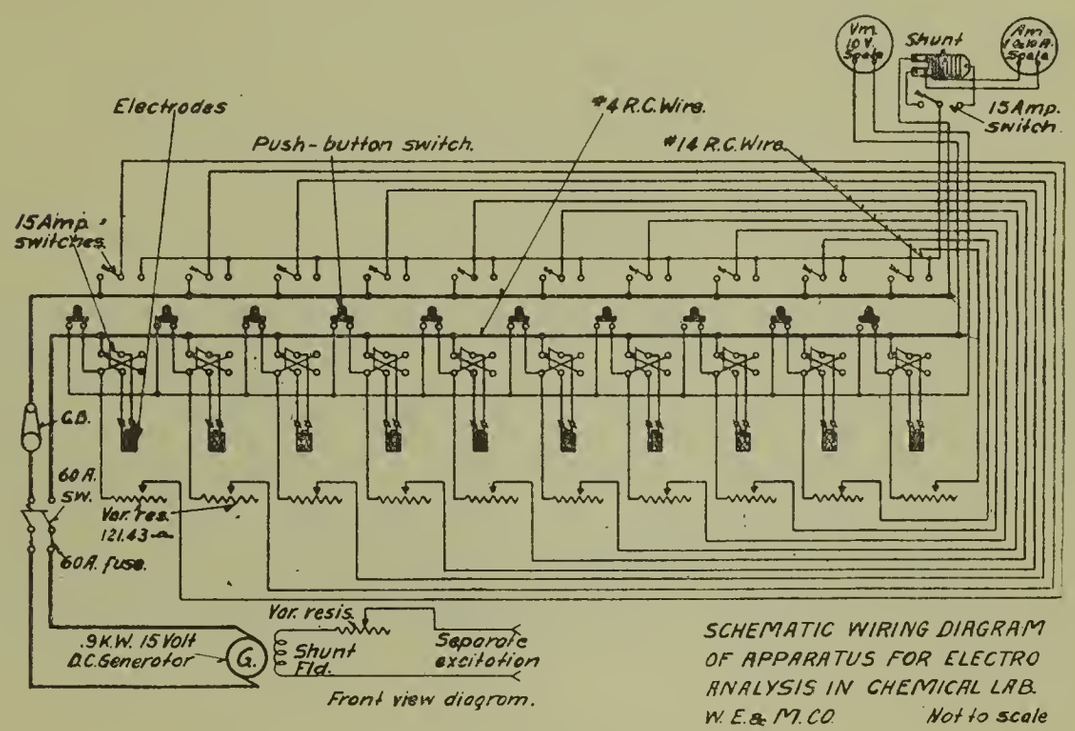

FIG. 3.

Current: The necessary current for the electrolysis is furnished from a motor-generator set that delivers 60 amperes at 15 volts at the terminals of the generator.

Resistance: Rheostats conveniently mounted enable the maximum and minimum currents required for analysis to be obtained. Many types of electro-analysis apparatus are so wired that they have the same current at each unit, but with our arrangement each unit has a separate rheostat, and a copper analysis may be run on one of them with 6 amperes and a babbitt metal on the adjoining set at 0.5 ampere.

Reversal of Polarity: The larger platinum cylinder is normally the cathode and the smaller one the anode, but the polarity may be changed at will by means of a double throw switch, the two positions being marked respectively, cathode and anode.

Wiring: A hardwood cabinet encloses the apparatus. The wiring is placed on the back of the cabinet and Fig. 3 shows it in diagram. Although injury to the apparatus from excessive current is not likely, it is guarded against by suitable fuse protection.

The design of the apparatus was made by Mr. F. P. Lauffer, mechanical engineer of the Research Division of the Westinghouse Electric and Manufacturing Co., aided by Mr. Edgar Cornell, Jr., alloy chemist.

DISCUSSION.

Mr. J. L. Jones: Since writing the paper we have devised a new stirrer, which is made of a rod of tungsten about 4 inches long—just sufficient to enable it to be held properly in the clamps and just long enough to extend above the solution where it is exposed to the corrosive spray. The balance of the rod is composed of glass. It is somewhat simpler than the one originally described in the paper, cheaper, and can be made in about half the time.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, Oct. 5, 1917, President Fink in the Chair.

THE SPECTROSCOPIC DETERMINATION OF SMALL AMOUNTS OF LEAD IN COPPER

By C. W. Hill and G. P. Luckey.*

[Abstract.]

For the rapid determination of lead in factory samples of copper, a known weight of copper is placed in a slight cavity in a lower positive graphite electrode, and a fixed or rotating carbon negative electrode used above. A grating spectroscope serves to observe the spectrum. The time required to cause the disappearance of the bright lead line in the spectrum, or to cause its enfeeblement to a certain dimness, is measured with a stop-watch. With carefully regulated arc, the times vary regularly with the amount of copper used and with its percentage of lead, *e. g.*, from 14 seconds with 0.2 g. copper containing 0.004 percent lead to 277 seconds with 1 g. copper containing 0.038 percent lead. The results are sufficiently reliable for practical use in the copper refinery.

The following experiments are the result of the need for a rapid method for the determination of small amounts of lead in copper for use in the factory control of copper refining. On account of delay in securing apparatus and the pressure of other work, the authors have not been able to conduct all of the desirable experiments incident to the problem, but are moved to publish the results of the work accomplished in the hope that others may find use for the method and so improve or modify it that it may be capable of broader application.

* Research Chemists, Westinghouse Electric & Mfg. Co., East Pittsburgh, Pa.

APPARATUS.

The apparatus used consisted of a vertical arc and a small grating spectroscope, together with suitable resistances and condensing lens, shown diagrammatically in Fig. 1. The arc was formed between an upper negative electrode of half-inch (13 mm.) carbon rod and a small sphere of copper resting in a slight cavity in the lower positive graphite electrode. The spectrum was formed by a two-inch (5 cm.) concave grating of three feet

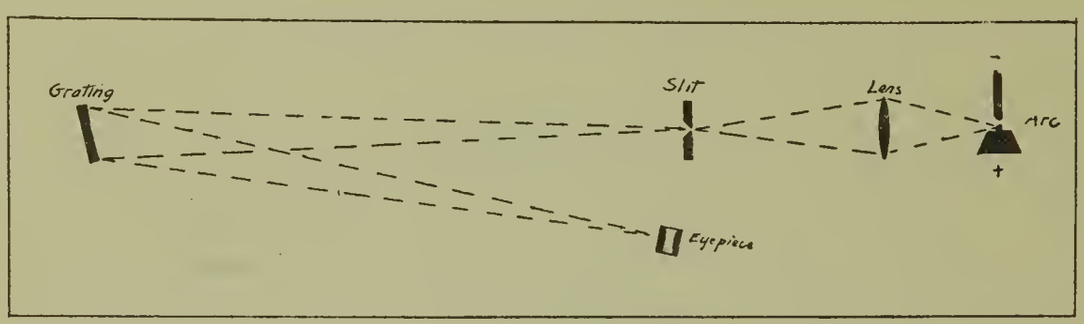

FIG. 1.

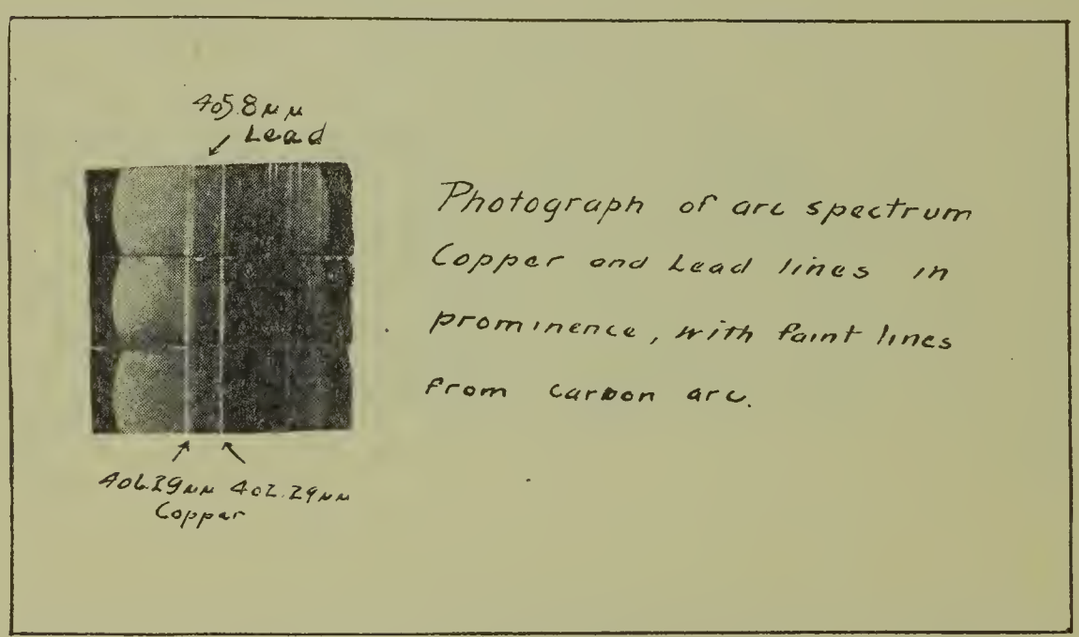

FIG. 2.

(90 cm.) focal length ruled 15,000 lines to the inch (6,000 per cm.). Lines of the first order spectrum were observed with an eyepiece magnifying ten times. For the determination of lead in copper, the brightest visible line in the lead arc spectrum (405.8 $\mu\mu$) was observed. This line lies in the violet between the two bright copper lines, 405.29 $\mu\mu$ and 402.29 $\mu\mu$. Fig. 2 shows the photograph of the spectrum as observed, with the lead and copper lines in prominence and the many fainter lines resulting from the carbon arc.

In later experiments, a rotating upper electrode was used. This steadied the arc materially and held the focus of the arc on the slit, so that the arc required no attention after starting.

PROCEDURE.

Weighed samples of copper shot of known lead content were subjected to an arc of constant length and current. By means of a stop-watch the time was measured from the melting of the sample until the disappearance of the lead line in the spectrum. The end-point was easily determined, since the lead line lies almost above the first line of a carbon band. At first the line appears very bright, but as the lead disappears the line assumes the same brightness as the remainder of the carbon band of lines. It is much easier to match the line in brightness than to observe the final disappearance of a line.

OBSERVATIONS.

Using the apparatus and method described, experiments were made using varying weights of copper samples (0.1 g. to 1.0 g.) containing different percentages of lead (0.004 to 0.216 percent). The arc was maintained at a length of 0.5 cm. and consumed 10 amperes on a 220 V. circuit. The results are tabulated in Table I and plotted in Figs. 3 and 4.

TABLE. I.

Time for Different Weights of Samples.

Percent of Lead in Sample	Time in Seconds for Weights of Samples Given					
	0.1 g.	0.2 g.	0.4 g.	0.6 g.	0.8 g.	1.0 g.
0.004	...	14	20	...	...	...
0.005	...	...	...	67	98	150
0.006	...	34	40	...	...	...
0.008	...	50	67	...	...	...
0.009	...	54	...	...	...	...
0.011	22	65	87	135	173	210
0.014	...	67	95	...	...	...
0.016	...	...	120	...	...	...
0.028	...	92	...	215	240	277
0.031	...	...	155	...	...	...
0.045	35	...	180	...	...	...
0.062	50	140	227	...	...	...
0.097	...	203	...	...	...	...

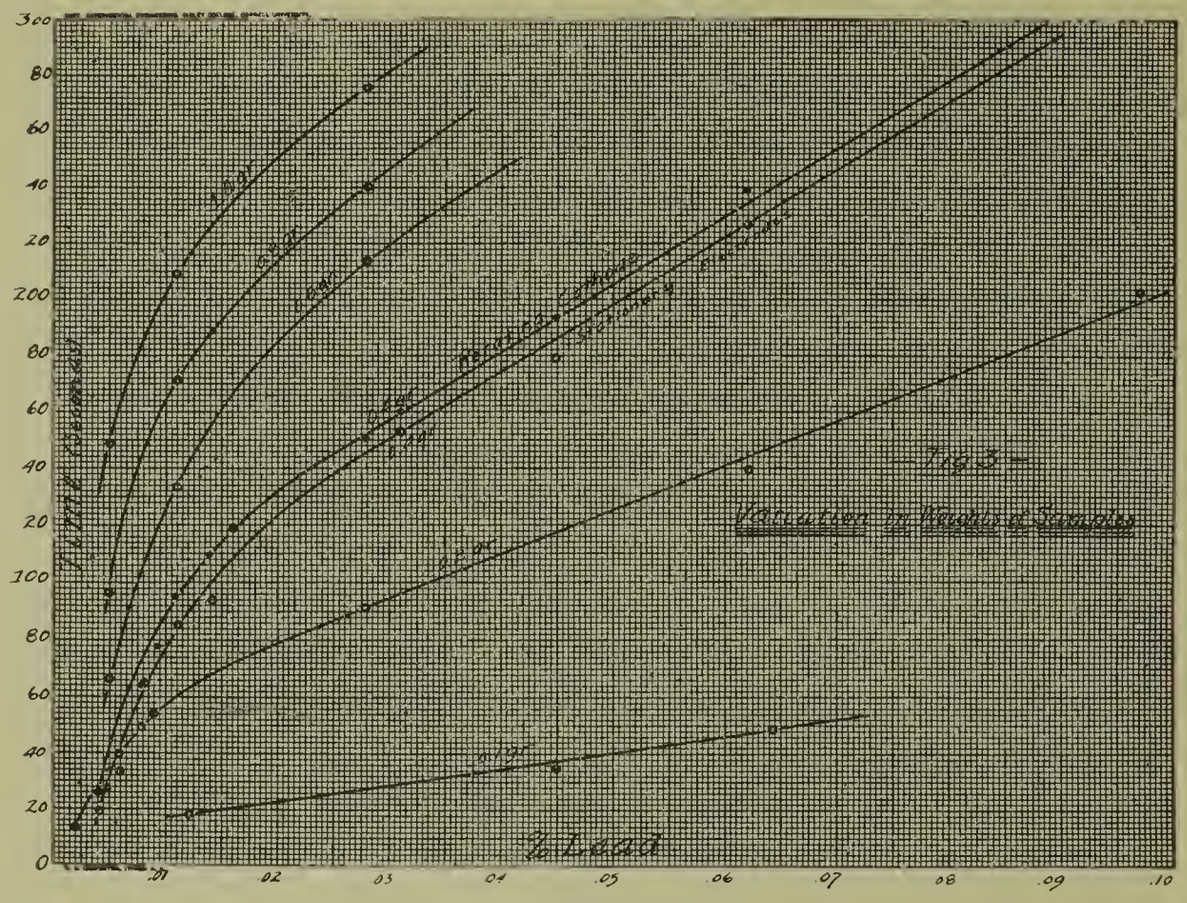

FIG. 3.

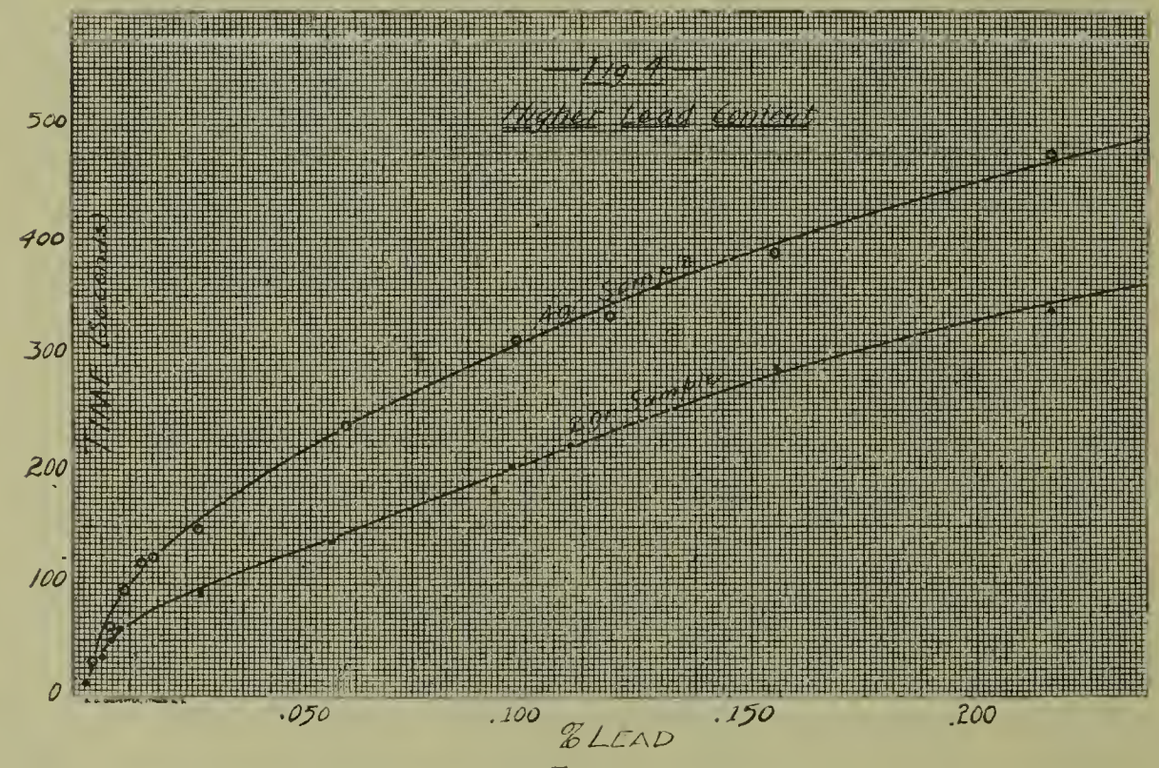

FIG. 4.

The size of sample which will give the best results is dependent upon the range of lead content to be determined and upon the size of electrodes and current conditions. With a given arc, larger samples increase the accuracy at low percentages of lead,

but lengthen the time of measurement for samples of high lead content. For our specific problem and arc arrangement, samples of 0.4 g. are the best.

Table II shows the results of two runs made several months apart, one determination being made with a stationary upper electrode and the other with a rotating electrode. Individual readings are given to show the deviation from the average. (Plotted in Fig. 3.)

TABLE II.

Check Runs With 0.4 g. Samples.

(Time in Seconds)

Percent Lead by Analysis	Stationary Cathode	Rotating Cathode			
		Averages	Individual Readings		
0.004	20	26	25	23	29
0.006	40	...	..	..	..
0.008	67	62	65	60	60
0.011	87	96	100	95	98
0.014	95	110	(From curve)		
0.016	120	120	120	120	120
0.028	146	151	146	155	154
0.031	155	160	(From curve)		
0.045	180	194	(" ")		
0.062	227	240	235	240	245
0.097	...	313	290	315	334
0.119	...	331	325	329	338
0.156	...	387	375	400	...
0.216	...	474	485	463	...

It is to be noted that the standardization of the method was made by use of factory samples the chemical analysis of which had been made with great rapidity and with the usual errors incident to the method. The readings were taken as a rule at random, with lead content unknown to the person observing the spectrum. Repeated runs by different observers showed variations of the same magnitude as those in Table II.

In using the method as a check on the refining process, it is not necessary to have a complete standardization curve. Samples of satisfactory material may be run from time to time, and the refining process continued until furnace samples show a disappearance time equal to or less than that of the standard sample. If

desirable, the disappearance time could be plotted against furnace operating time.

For factory use it is proposed to use a suitable spectroscope, if one can be obtained which has sufficient diffraction to show the lines distinctly. A Joly balance will be substituted for the usual analytical balance, since it is sufficiently accurate and is better adapted to factory usage.

CONCLUSIONS.

From the above experiments it is concluded that the empirical method outlined permits the rapid determination of small amounts of lead in copper. It is necessary to maintain constant conditions of arc and to standardize the apparatus against known samples. Several check readings should be taken when considerable accuracy is desired.

For control work the time of disappearance of the lead for a satisfactory sample of copper may be used as a standard, and the refining process carried on until the furnace sample shows an equal time of disappearance.

OTHER APPLICATIONS.

The use of the arc is limited to the determination of small amounts of impurities which are volatilized at a comparatively low temperature in the presence of a metal which is not rapidly volatilized at a comparatively high temperature and which gives a steady arc, the existence of strong characteristic spectrum lines being assumed. (In the present case the above statement should probably be modified so as to apply to the oxides of the metals rather than the metals alone.) Some metals would give better results in an inert atmosphere.

The method might be applied to metals of low boiling point or even to salts, by substituting a heating strip in place of the arc and securing the arc or spark spectrum from the vapors arising from the heating strip.

Research Laboratory,
Westinghouse Electric & Mfg. Co.

DISCUSSION.

W. R. MOTT[1]: It is a pleasure to know that such accurate quantitative analyses can be made by this method, similar to that which I have found convenient in a less refined form for the study of fractional distillation of alloys. The average deviations of the time readings appear to be ± 6 percent, which is very good accuracy referred to small amounts of lead in copper.

The method as used by the authors appears likely to be best worked out with metals forming molten alloy beads not wetting the carbon. In my analytical outline using arc images, these easily reduced metals not wetting carbon were called Class A, and were characterized also by giving little or no arc shell. The more common of these metals are arsenic, cadmium, zinc, thallium, lead, bismuth, antimony, silver, copper, tin and gold. The boiling points of these may be stated to be about as follows:

	° C.	
Arsenic (sublimes)	554	
Cadmium	778	
Zinc	918	
Thallium	1306	(Wartenberg)
Bismuth	1420	(Greenwood)
Antimony	1440	(Greenwood)
Lead	1525	(Greenwood)
Silver	1955	(Greenwood)
Tin	2270	(Greenwood)
Copper	2310	(Greenwood)
Gold	2530	(Greenwood)

The combinations of the lower boiling metals with the higher boiling metals offer the best opportunity of getting combinations parallel to those of lead and copper. However, like mixtures of water and hydrochloric acid, certain metals give combinations having a higher boiling point than that of either metal alone. These exceptional cases are arsenic and cadmium, antimony and copper, tin and copper, and mixtures of bismuth, antimony and lead. In these cases spectrum examination would not show a disappearance of either constituent. By examining the upper and lower deposits in carbon cups, I have discovered several very delicate tests capable of semi-quantitative valuation for many of these and other elements. For example, 100 g. of lead with 1/50

[1] Research Laboratory, National Carbon Co., Cleveland, Ohio.

mg. of silver easily shows a beautiful purple upper deposit characteristic of silver. Tin with minute amounts of gold gives a very delicate test by forming Purple of Cassius. The details of these and numerous other tests I hope to present at a later meeting of our Society. Again let me express my high regard for the interesting work of Dr. Hill and Mr. Luckey.

M. G. Lloyd[2]: A spectroscopic method of quantitative analysis has been in use at the Bureau of Standards, Washington, for determining the amount of impurities in tin boiler plugs. A concave grating is used to form a spectrum of the spark, and this spectrum is photographed with an exposure of five minutes.

The spectra are compared with those of samples whose contents are known by means of chemical analysis, and the results are quite as reliable as the chemical analysis, when the amount of impurity being sought is only 0.1 percent. The quantity is determined by the intensity of the line in the photographed spectrum. An analysis of ten plugs for four impurities may be easily made in 1½ hours by a single observer. The principal impurities are lead, zinc, copper and iron.

The photographic method has the advantages of a permanent record, and the ability to make the examination for several elements at once.

Quantitative spectroscopic analyses of steel have also been made, especially for the determination of chromium and titanium. For such elements as niobium and molybdenum, the estimation of small quantities is more reliable than by chemical analysis.

C. W. Hill (*Communicated*): The discussion of Mr. Mott would seem to be pertinent, and should be borne in mind in attempting to apply the method to other elements than those which we have used.

A word of caution should be said regarding the method described by Mr. Lloyd. It may apply in certain cases, using the spark, but it does not apply to the arc spectrum in any of the cases which we have studied. Before developing the method which we have discussed, we made a large number of tests in which we attempted to connect the intensity of the lead line with the percentage of lead in the sample. It is extremely difficult to

[2] Bureau of Standards. Washington, D. C.

estimate the strength of a line in any quantitative manner. It would appear that the intensity of the lead line in the arc spectrum in our case depends upon the rate at which the lead is being expelled from the copper. This would in turn depend upon the vapor pressure of the lead, which is influenced by the temperature. With given size of samples and uniform electrical conditions, we found a qualitative difference in the intensity of the lead line with content of lead, but were unable to estimate it accurately even by use of photographs. Therefore, while the method of Mr. Lloyd may apply to the spark spectrum, it is our opinion that it would not be practical, if it is possible, with the arc spectrum.

Since the paper was written we have succeeded in using a spectroscope with two prisms in place of the grating. A commercial form of the apparatus may be described at a later date after it has been thoroughly tested under factory conditions.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

SYNTHETIC GASOLINE BY ELECTROCHEMICAL MEANS

By Louis Bond Cherry.*

[Abstract.]

Description of passing mixtures of heavy hydrocarbon vapors and natural gas through electrically-heated tubes, and subjecting them therein to the electrical discharge of a high-tension, high-frequency oscillating current. Results show the conversion of 40 to 70 percent of the heavy hydrocarbon into gasoline. The laboratory apparatus is described, next the first large experimental plant, and lastly a commercial sized unit, which is now being assembled.

This account of experiments in the production of synthetic gasoline, by electrochemical and electrothermal means, will probably be of interest on account of the growing scarcity and ever increasing demand for motor fuels, and also on account of the novelty of the process.

To announce that the gasoline problem had been solved would be too startling for the average scientist to believe without knowing how it was accomplished, and to say that it was done by electrochemical means would at once create a group of "Doubting Thomases." But, to first present a theory and then follow with an account of experiments supporting the theory, will be most convincing if the resultant account of the experiments justifies the theory.

It is well known that most of our crude oil distillates and natural gases of the United States are of paraffin series of hydrocarbons, conforming to the formula C_nH_{2n+2}, and that natural gas is largely composed of methane (CH_4), which occupies a place at the beginning of the series and is the simplest of the group; while going toward the more complicated molecular structures we find about

* Electrochemist and Electrical Engineer.

sixty well defined, saturated, normal hydrocarbons, terminating with the solid hexacontane ($C_{60}H_{122}$), with a melting point of 101° C.

Our commercial gasoline occupies a place in the series a little above those which are normally gas, and is composed of a probable blend of hexane (C_6H_{14}), heptane (C_7H_{16}), and octane (C_8H_{18}), for a good grade of gasoline, although we are tempted to believe some grades of gasoline on the market today contain hydrocarbons of much higher boiling points.

The above mentioned hydrocarbons are called paraffins because they resist at low temperatures the action of most reagents, and are, in fact, compounds with an almost indifferent character, refusing to take on more hydrogen or even to chemically unite with any of their associates.

If we *could* chemically unite, in proper proportion and form, sufficient methane, or hydrogen gas, to certain of the heavier hydrocarbons, we could produce synthetic gasoline.

The amount of gas necessary to do this is surprisingly small, as will be seen from the following table of the paraffin group which shows the percentage, by weight, of carbon and hydrogen in the different steps of the series:

Name	Symbol	Mol. Wt.	Percent C (by wt.)	Percent H (by wt.)
Methane	CH_4	16	75.00	25.00
Ethane	C_2H_6	30	80.00	20.00
Propane	C_3H_8	44	81.81	18.19
Butane	C_4H_{10}	58	82.76	17.24
Pentane	C_5H_{12}	72	83.33	16.67
Hexane	C_6H_{14}	86	83.72	16.28
Heptane	C_7H_{16}	100	84.00	16.00
Octane	C_8H_{18}	114	84.21	15.79
Nonane	C_9H_{20}	128	84.38	15.62
Decane	$C_{10}H_{22}$	142	84.51	15.49
Undecane	$C_{11}H_{24}$	156	84.62	15.38
Dodecane	$C_{12}H_{26}$	170	84.71	15.29
Tridecane	$C_{13}H_{28}$	184	84.78	15.22
Tetradecane	$C_{14}H_{30}$	198	84.85	15.15
Pentadecane	$C_{15}H_{32}$	212	84.91	15.09
Hexadecane	$C_{16}H_{34}$	226	84.96	15.04
Heptadecane	$C_{17}H_{36}$	240	85.00	15.00
Octodecane	$C_{18}H_{38}$	254	85.04	14.96
Nonodecane	$C_{19}H_{40}$	268	85.07	14.93

The small increase in percentage of hydrogen necessary to convert some of the heavier fractions of crude oil into the lighter and lower boiling point compounds is clearly shown. For example, heptane (C_7H_{16}) contains only 0.51 percent more H than decane ($C_{10}H_{22}$).

On account of the cost of producing hydrogen, natural gas (which is composed mostly of methane, CH_4), was suggested as being the most desirable, and the following equations illustrate the possibilities of using methane gas (CH_4) to combine with the heavy, high boiling point hydrocarbons, to obtain low boiling point products, if it were possible to obtain reactions as herein illustrated.

Nonane ..... $C_9H_{20} + 2(CH_4) = 2(C_5H_{12}) + CH_4$
Decane ..... $C_{10}H_{22} + 2(CH_4) = 2(C_6H_{14}) + H_2$
Undecane ..... $C_{11}H_{24} + 2(CH_4) = 2(C_6H_{14}) + CH_4$
Dodecane ..... $C_{12}H_{26} + 2(CH_4) = 2(C_7H_{16}) + H_2$
Tridecane ..... $C_{13}H_{28} + 2(CH_4) = 2(C_7H_{16}) + CH_4$
Tetradecane ..... $C_{14}H_{30} + 4(CH_4) = 3(C_6H_{14}) + 2H_2$
Pentadecane ..... $C_{15}H_{32} + 3(CH_4) = 3(C_6H_{14}) + H_2$

After studying the electromagnetic theory of matter it was conceived, that, if the only difference that existed between carbon and hydrogen (or between two hydrocarbons such as methane, CH_4, and nonane, C_9H_{20}), was the number and organization of infinitesimal units of electro-radiant energy which we are pleased to term electrons, and that if we mechanically mixed the vapors of a hydrocarbon, such as nonane (C_9H_{20}), with methane (CH_4), or natural gas, and impressed upon this mixture electro-radiant energy of the proper periodicity, tone, or keynote, we would be able to break up the "electronic solar system" of the molecules and cause them to vibrate or "dance to new music," and on leaving the zone of electrical action they would assume some natural organization and periodicity depending, to some extent, on the frequency of the energy impressed upon them, and on physical conditions, such as temperature and pressure.

The first experimental equipment was a small laboratory affair, the still being composed of a common piece of 3 in. (7.5 cm.) pipe, the ends of which were closed with caps, threaded and screwed on. The reaction tubes wherein the gas and oil vapors were subjected to a high-voltage oscillatory high-frequency current, were

made of 1 in. (2.5 cm.) pipe which had been plugged and reamed, the ends of which were fitted with modified spark plugs which served to lead in the current through a fine No. 30 (0.25 mm.) platinum wire, which wire passed through the center of the pipe from end to end, and which, when connected to the high-frequency coil, subjected the gas and oil vapor, passing through the pipe, to a silent electric discharge, alternating back and forth, between the central electrode and the pipe, and at right angles to the direction of the flow of gas.

As above mentioned, this was a very crude equipment, without pyrometers or pressure gauges, and was operated at high pressure and high temperature, but it served to demonstrate the feasibility of the process, by converting 42½° Bé. kerosene into 65° Bé. gravity gasoline. As above stated this was done at high pressure and temperature, and was operated for several hours before the equipment exploded, completely wrecking the small frame building in which it was located.

The C. & C. Developing Co., a corporation, was then organized, a part of the capital stock of which was sold to raise funds for the purpose of carrying on experiments toward perfecting equipment which would operate commercially in a satisfactory manner. Experiments proved that a very high frequency was necessary to obtain low boiling point compounds, and that increasing the frequency, would raise the gravity (Bé), and lower the boiling point. In the building of this larger equipment an attempt was made to use wireless telegraph equipment, but as such apparatus is designed only for intermittent service, it proved inadequate, and we concluded to design special equipment, photographs of which are herewith shown.

This plant was designed to convert one gallon of oil per minute. The still was of pipe construction, 4 in. (10 cm.) double-extra-heavy pipe, welded flanges, electrically heated, and consumed about 40 K.V.A. of electrical energy at 60 volts, using 60-cycle alternating current.

Electric power was brought in at 110 volts, and by means of an autotransformer, arranged with 25 three-volt taps connected to a switchboard panel equipped with a special rotary switch with 25 contact points or clips (see figure No. 2), control of the

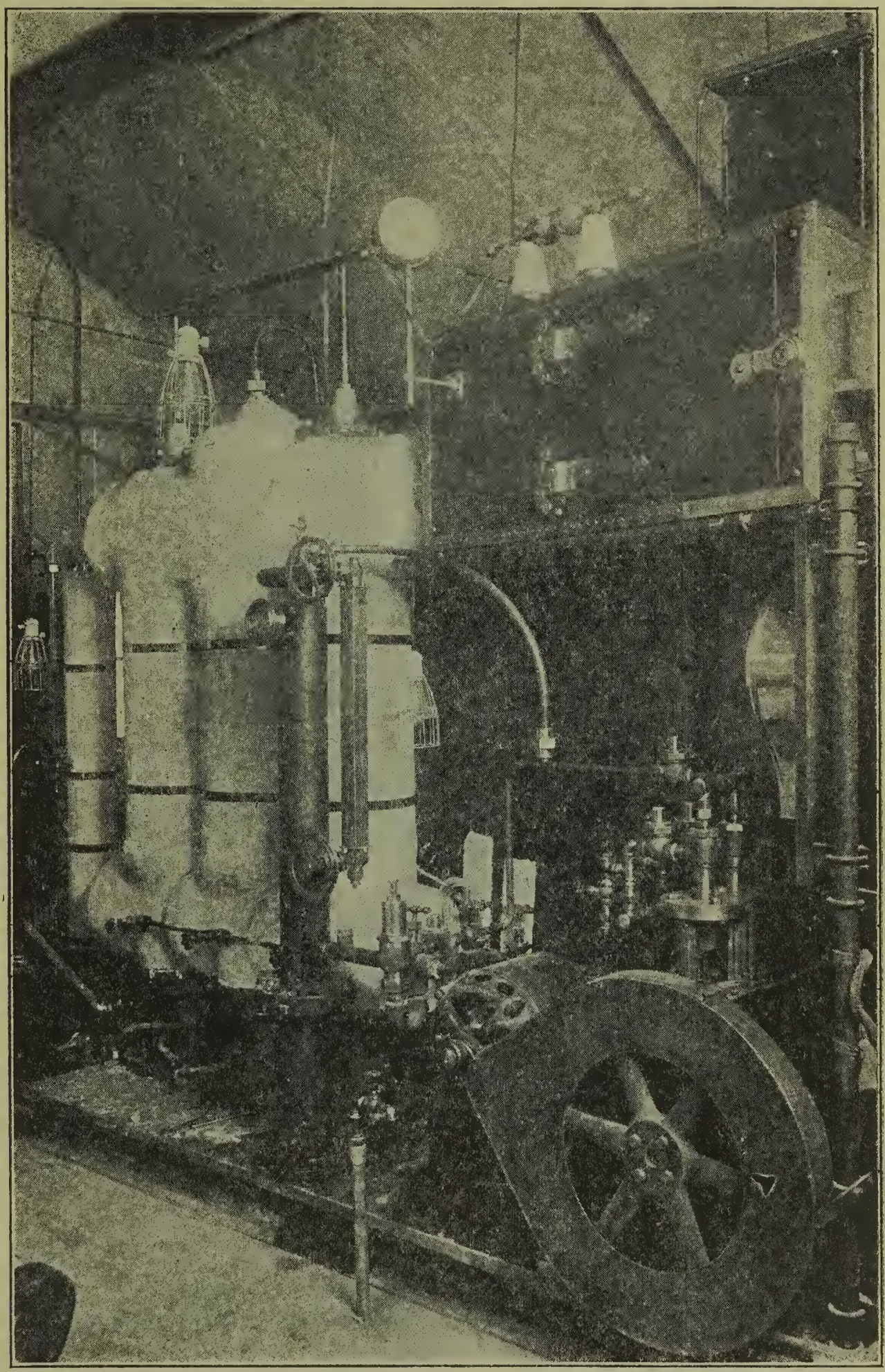

FIG. 1. First Experimental Plant.

heat was very sensitive. The oscillatory electrical generating apparatus for this plant consisted of 1 K. V. A., 110 to 25,000 V, step-up transformer, with a variable reactance in the primary, a variable condenser, rotary spark-gap, and oscillatory transformer.

The operation of this plant was interrupted several times by

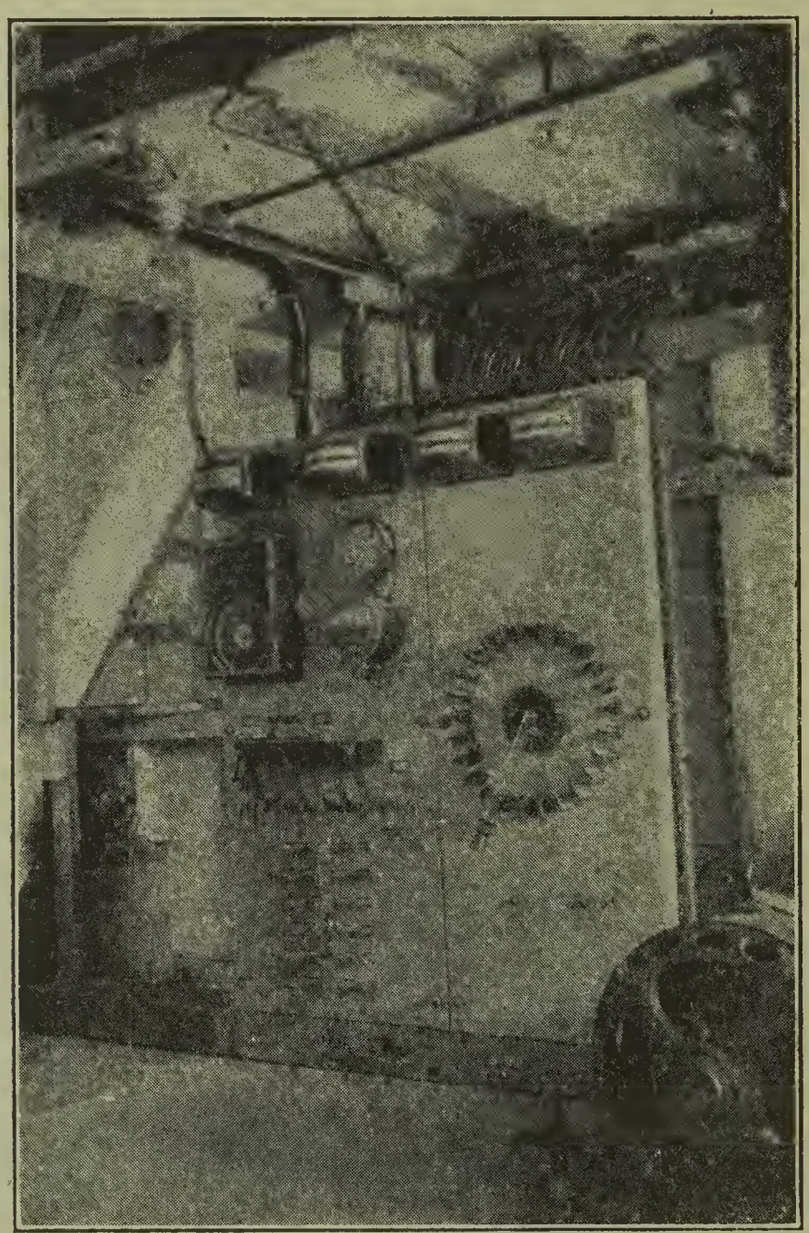

FIG. 2. Switchboard of First Plant.

leaks developing at the flanged joints of the pipe still. These flanges were gasketed with corrugated sheet copper, and due to the expansion, strain under heat, and high pressure, this kind of gasket proved inadequate, and after much costly experimenting, which included a change to heavier flange bolts, a gasket was

secured made of woven asbestos and spun over with sheet copper, which proved very efficient.

As stated above, this plant had an evaporating capacity of one gallon per minute and when running on kerosene of 42° Bé. density (0.81) and natural gas, and at 75 lb. working pressure per sq. in. (5 atmospheres), 56 percent of the kerosene was converted into distillates having an initial boiling point of 45° C., and an end point of 160° C., with an average gravity of 56.3° Bé. (0.751).

The length of the reaction tubes of this plant was 3 feet (90 cm.), and the voltage of the high-frequency equipment was about 40,000, as measured by a sphere gap, with sphere 3 inches (7.5 cm.) in diameter.

The difficulties encountered with operation under high pressure were so great that it was decided to carry on experiments under atmospheric pressure, at which it was found that as good products could be obtained as when operating under pressure, but the percentage of conversion was not as great, and the gravity was not quite as high, 33 percent of the kerosene being converted, to an average density of 54° Bé. (sp. g. 0.761) and by re-running the residue (which was practically the same density as the original kerosene), it was discovered that an additional conversion of 33 percent was obtained for every time fractionation was made and the remaining residue run through the still.

The performance of this plant seemed to justify the following conclusions: (1) That the voltage and frequency of the treating current were not sufficiently high; (2) That the gas and oil vapors were not under treatment for sufficient length of time, in other words, the reaction tubes were too short; (3) Insufficient superheating of the oil and gas vapor might account for a part of the inefficiency.

At the conclusion of these experiments it was decided to build a larger plant and incorporate into its design the benefit of our experience with the old one.

To design high-frequency apparatus to produce current of several hundred thousand cycles per second, a potential upwards of a hundred thousand volts, current generating capacity of three to five amperes at the above voltage, and to build the equipment

so reliable that it could be operated continuously twenty-four hours per day and seven days per week, seemed a task so colossal and fraught with such difficulties, that many engineers predicted failure.

There was apparently only one policy to adopt in the design of

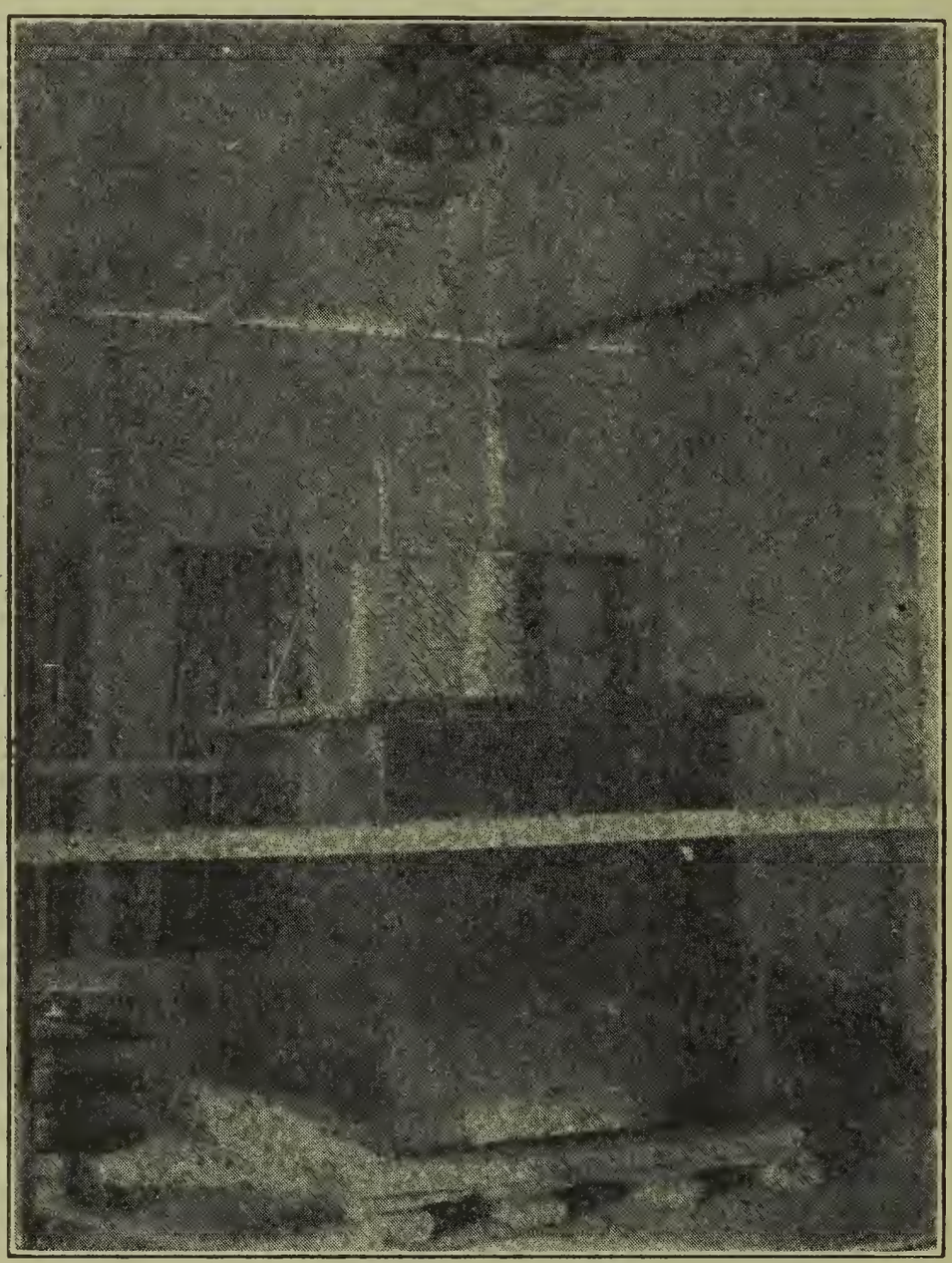

FIG. 3. 110/220—50,000/100,000 Volt Step-Up Transformer in Second Plant.

this equipment to meet the above service conditions, and that was the sacrifice of efficiency of operation for a large factor of safety in the electrical insulation.

Another thing that had to be avoided in order to obtain the extremely high periodicity necessary, was high resistance of the oscillatory circuit.

The step-up transformer was designed of the closed-core type, 110/220—50,000/100,000 volts, and rated at 30 K. V. A., but with a real capacity of 75 K. V. A.

The transformer coils, both primary and secondary, are of the pancake type, ribbon-wound, which allows free circulation of oil for cooling and insulation, and the secondary or high voltage coils are made up of ribbon which is comparatively very wide and thin, which has the effect of counteracting the self induction of the turns in the coil by electrostatic capacity. This is of marked advantage where extra high frequency is desired, in that it decreases

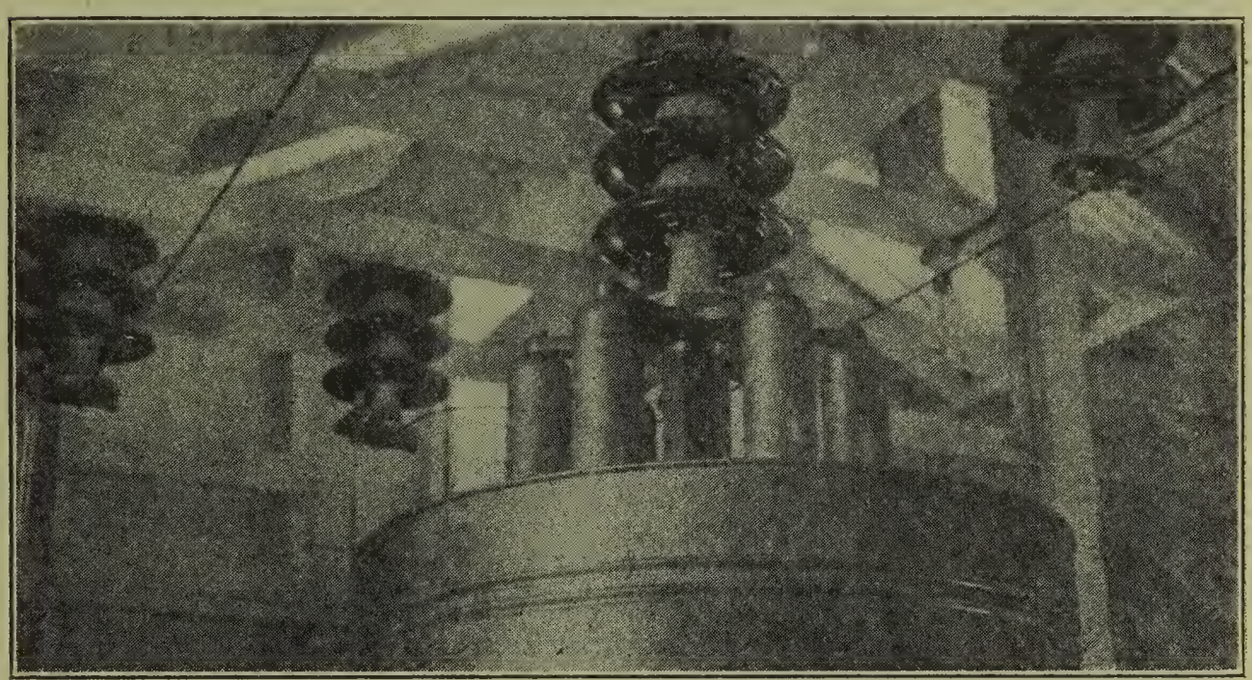

FIG. 4. Variable Tubular Electrostatic Condenser.

the time required at which the condenser will charge, and determines to a marked degree the periodicity of the discharge across the spark gap.

The fixed condenser was made with polished plate glass ¼ in. (6 mm.) thick, for the dielectric, four of these plates being used between each pair of copper plates of opposite polarity, and each glass plate was separated from the others by wood spacing strips ⅛ in. (3 mm.) thick, which was also placed at the ends.

This condenser was placed in a tank and filled with mineral seal oil which had previously been dehydrated and filtered.

This condenser was connected in a multiple with a tube-type variable condenser using 7 porcelain tubes, 40 in. long (102 cm.)

by 6¼ in. (16 cm.) outside diameter by 4¼ in. (11 cm.) inside diameter, immersed in mineral seal oil, as a dielectric, and seven 3½-in. (9 cm.) brass tubes 34 in. (87 cm.) long, connected (in symmetrical arrangement) to a brass spider at the upper end. The complete unit was supported by a high-tension insulator and was capable of being raised and lowered in the porcelain tubes by means of a rope, pulley, and counter-weight equipment.

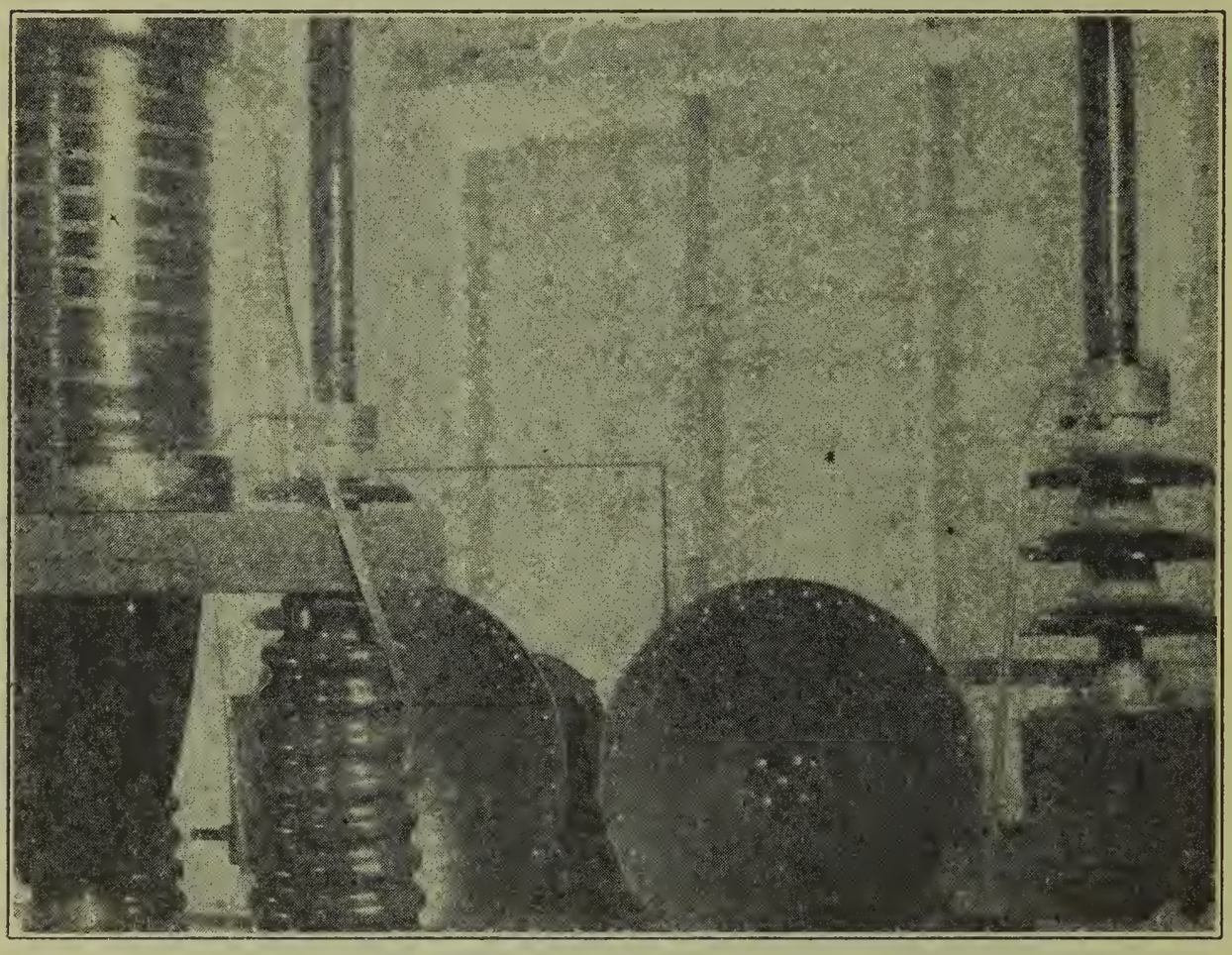

FIG. 5. (Right) Rotary Spark Gap. (Left) Lower Part of Oscillatory Transformer.

The outside or opposing element was made up of split copper tubes, the upper ends of which were connected to a heavy brass spider, so formed that the upper edges curved outward and away from the upper end of the copper tube, and the porcelain within it. This outward curving has the effect of lowering the potential gradient at this point, thus reducing the tendency to puncture the porcelain.

The spark-gap consists of two 20-in. (51 cm.) Bakelized fiber discs, directly mounted on extended shafts of two ⅓ H.P., 1,750

R.P.M. induction motors, which run in opposite directions. On the periphery of each disc was mounted a brass rim with a ½-in. (13 mm.) face.

The motors were mounted on guides, the bases of which were connected with a right- and left-hand screw, which served to regulate the gap between the discs, which were so placed that

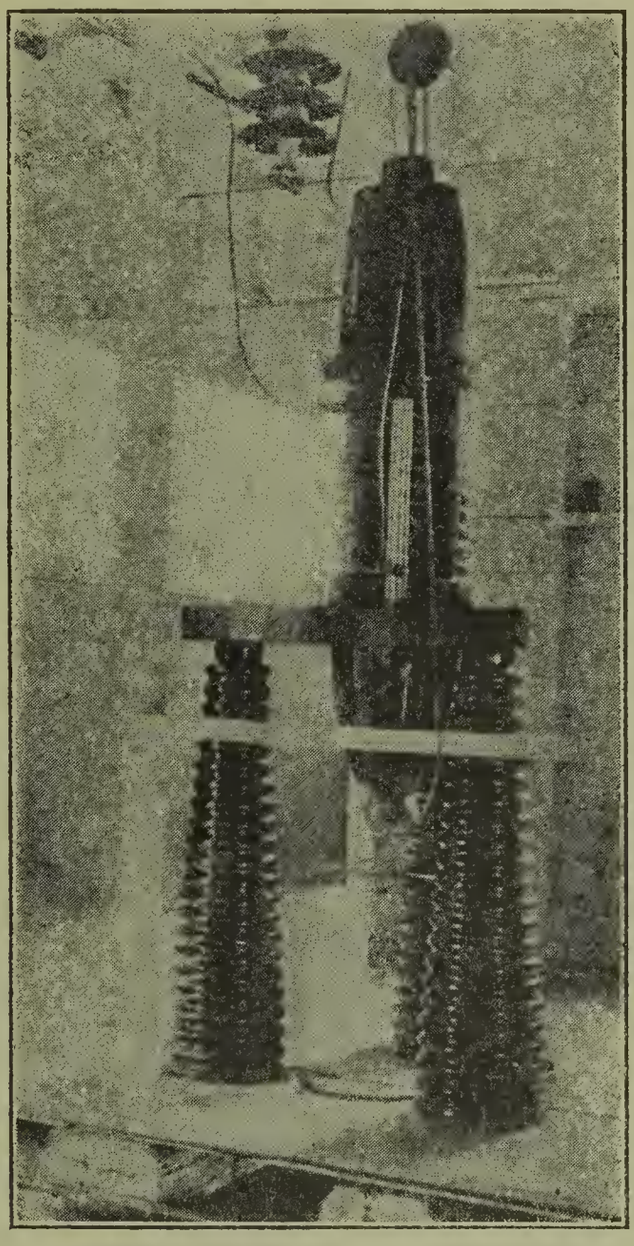

Fig. 6. Oscillatory Transformer.

they would rotate in opposite directions within the same plane, thus producing a strong air draft upward. Current was led to the two discs by fixed gaps on the opposite side from the main gap of each disc. This type of spark gap proved very efficient when operating on a 60-K.V.A. load at 100,000 volts, at which it will operate continuously.

In the design of the oscillatory transformer it was decided to use a vertical transformer case composed of porcelain, four feet (1.2 m.) in height, fourteen inches (35 cm.) greatest outside diameter, and with a wall one inch (2.5 cm.) thick. This case

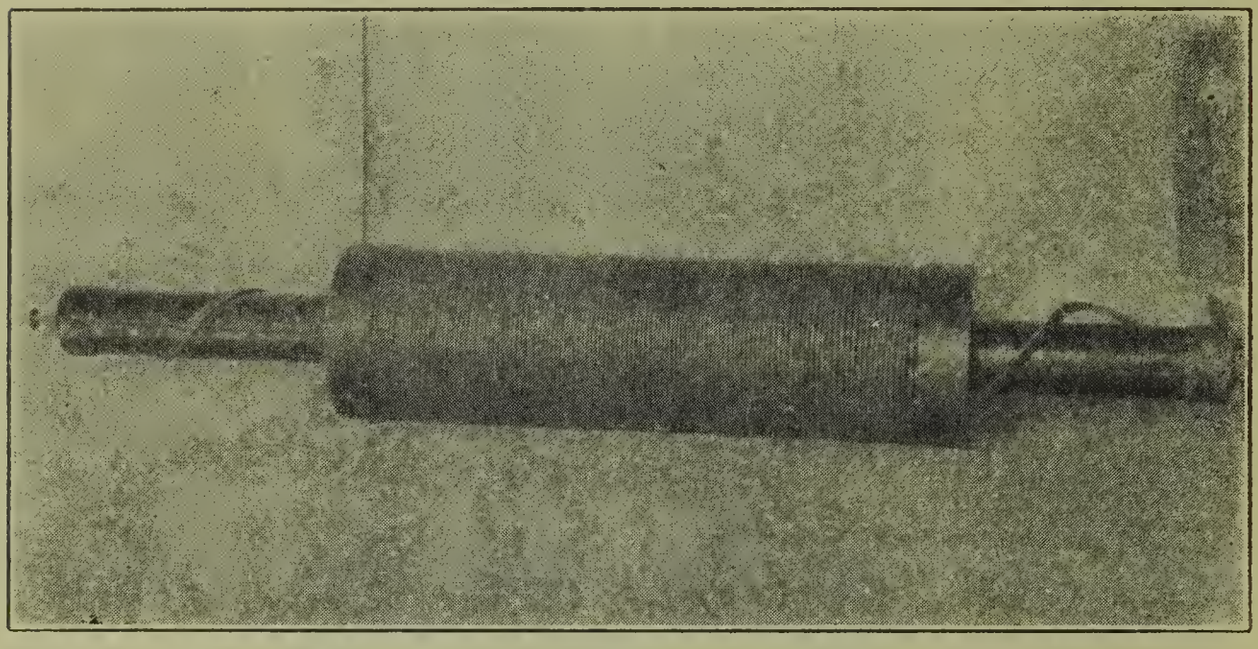

FIG. 7. Secondary Coil of Oscillatory Transformer.

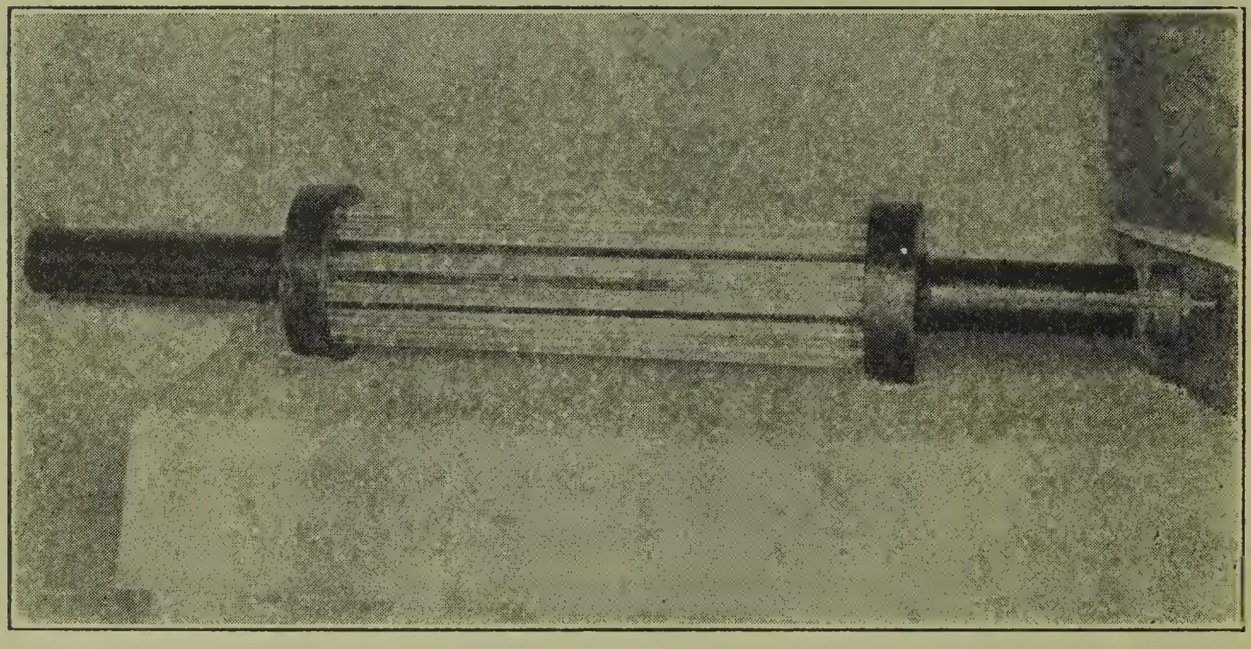

FIG. 8. Core of Secondary Coil of Oscillatory Transformer.

was supported vertically on three porcelain pillar insulators, 36 inches (92 cm.) in height. The primary winding consisted of ten turns of very flexible, braided, flat copper conductor, wound around the outside center of the transformer case.

The secondary winding was composed of 110 turns of the same conductor wound edgewise and insulated with four turns of Empire tape, the terminals of the coil being brought out at the two opposite ends of the transformer case. This coil is supported on a core, constructed of boro-porcelain tubes, terminating in wooden

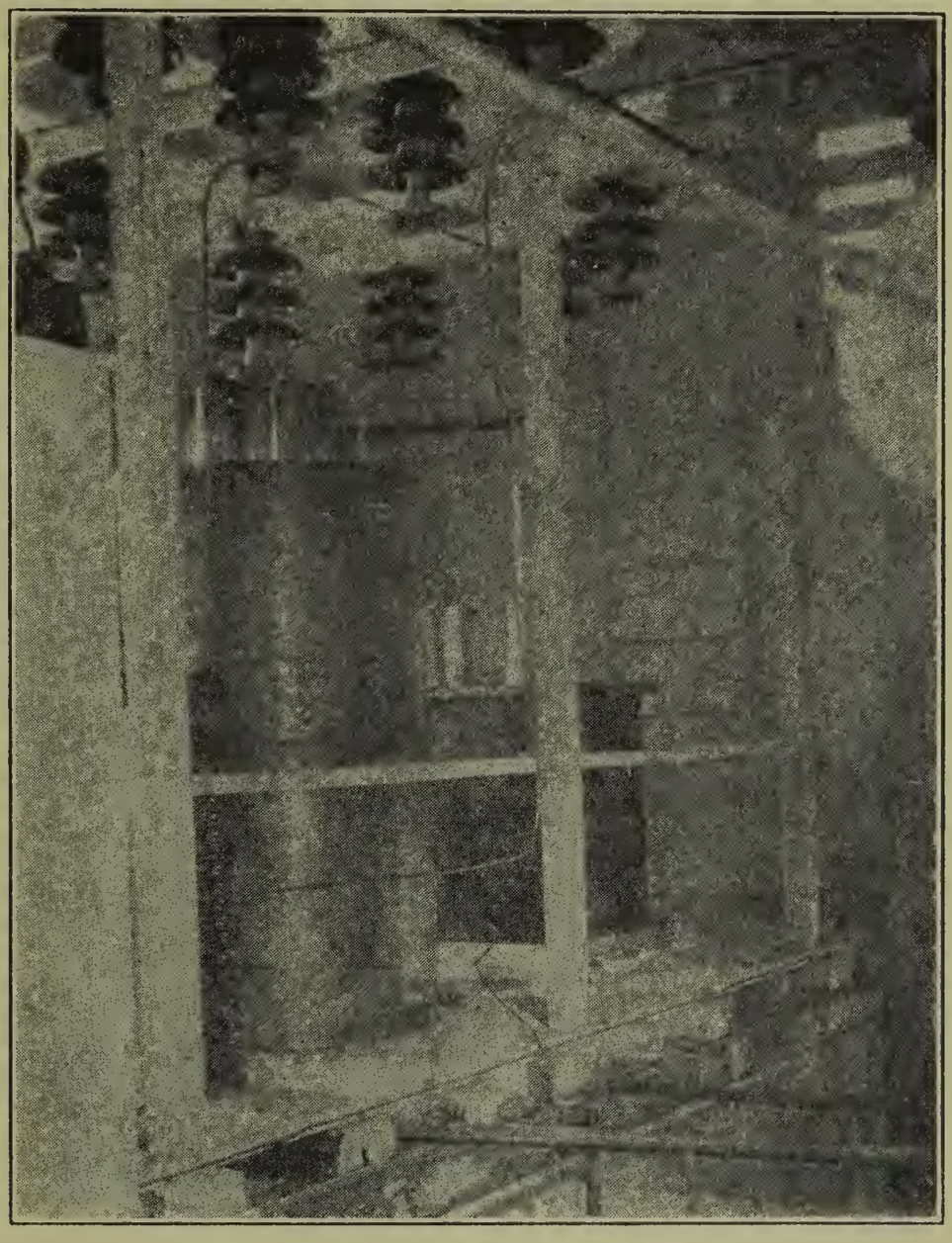

FIG. 9. High-Frequency Equipment.

spool ends, designed squirrel-cage fashion, and mounted on a 3-in. (7.5 cm.) porcelain tube which rests in the bottom of the transformer case, the upper end terminating in the cover. After being placed in position the case is filled with mineral seal oil, dehydrated and filtered.

This high-frequency equipment is mounted on a gallery about 8 feet (2.4 m.) above the main floor, and is capable of the widest range in operation, being capable of producing high-frequency current from 15,000 to about 250,000 volts, and has a capacity of converting into gasoline about 60,000 gallons of oil in one day of 24 hours.

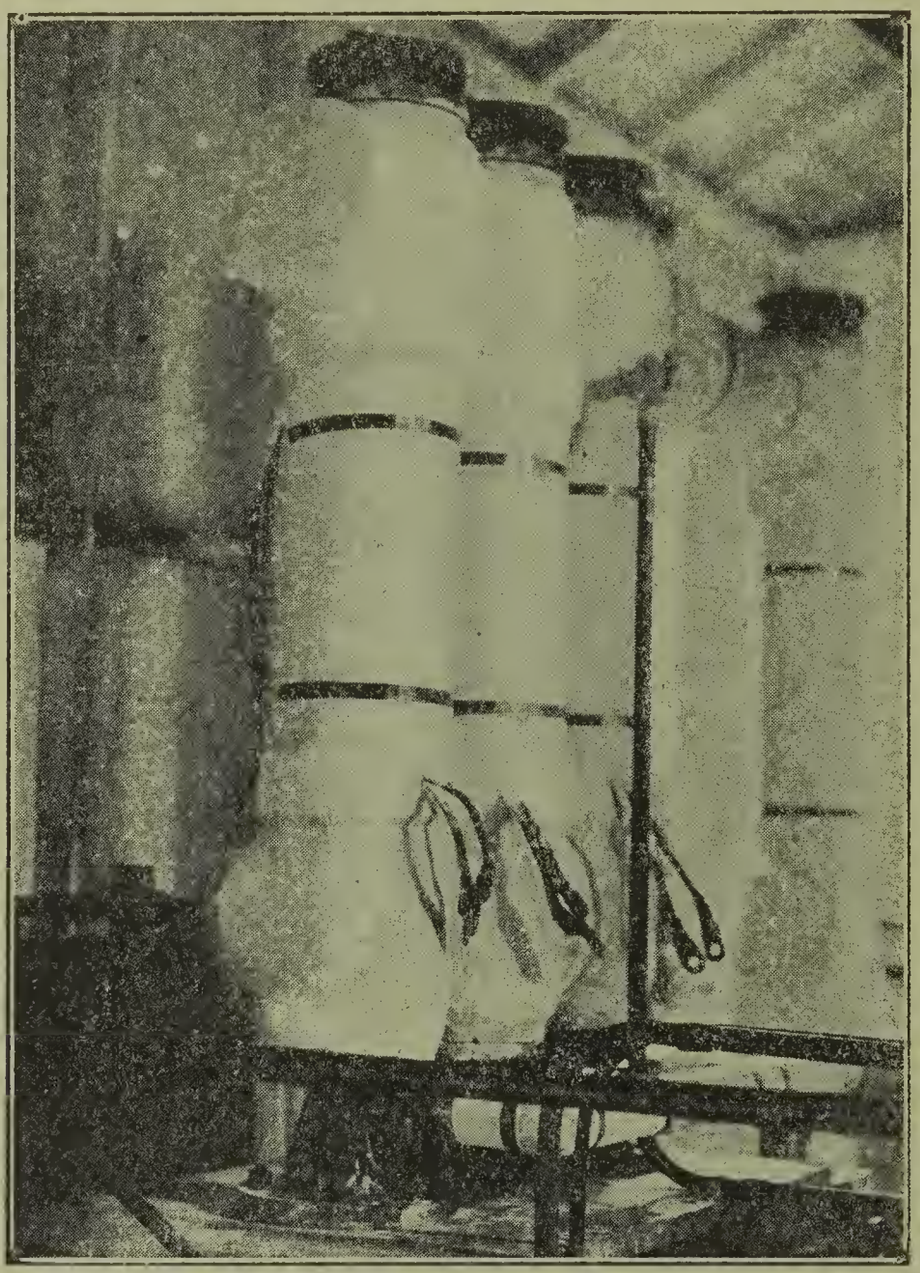

FIG. 10. Ten-Foot Reaction Tubes.

Experience with the first plant with its 3-ft. (0.9 m.) reaction tubes proved they were not sufficiently long, so in building this second plant the reaction tubes were built with a total length of ten feet (3 m.). For experimental purposes a seven-barrel still is used, which is fired with natural gas, and the vapor outlet of the still is connected to the reaction tubes.

These reaction pipes are heated by induction, being wound their entire length with double 0 copper cable (9 mm.) electrically insulated with a special fireproof insulating compound, and connected to an autotransformer similar to the one used in the first plant.

On operating this equipment, a 78 percent conversion was made from kerosene, and as this was not considered ideal, it was again decided to increase the length of the reaction tubes to 34 feet

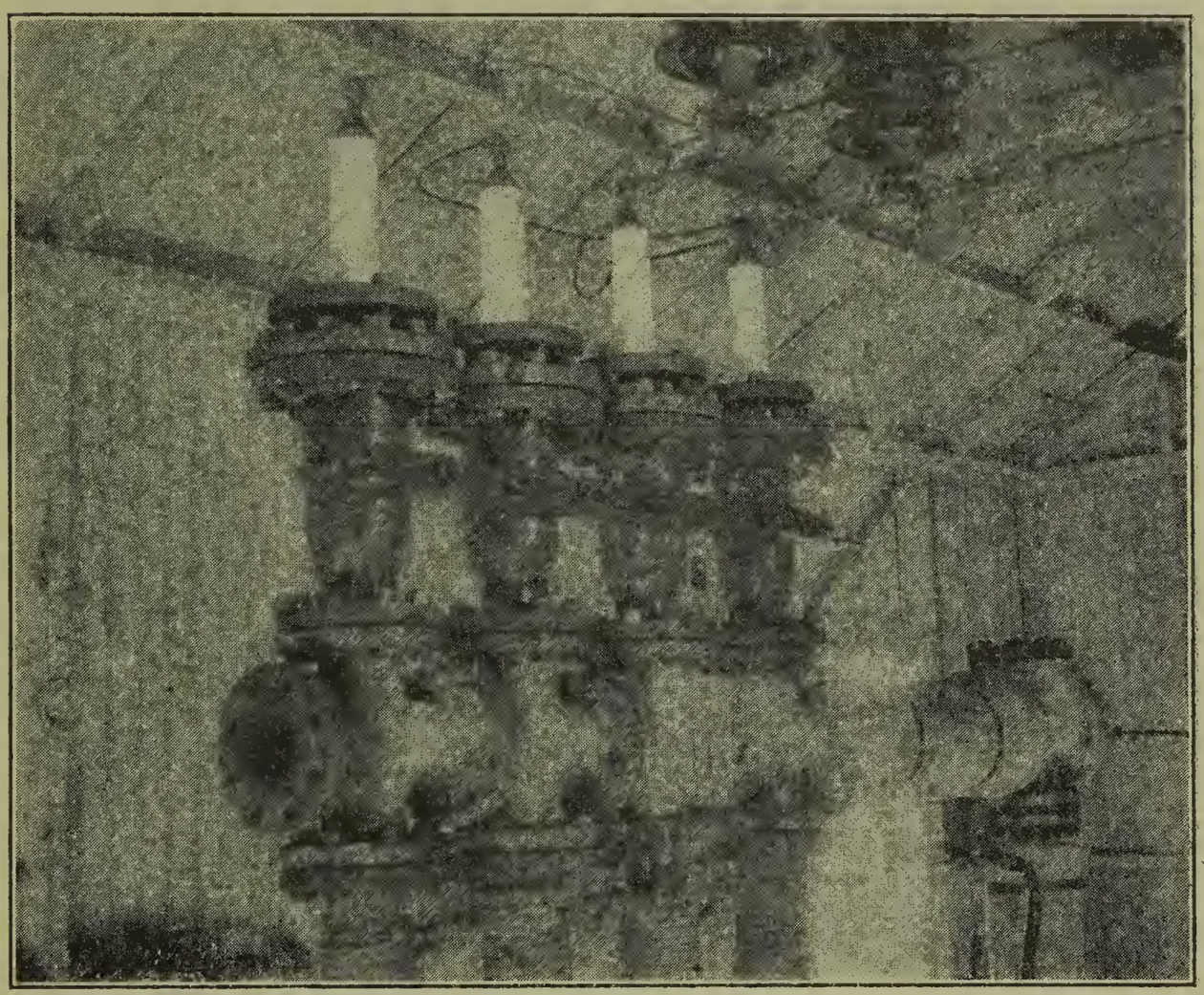

FIG. 11. Upper Part of 34-Foot Reaction Tubes.

(10.4 m.), which should give an efficiency approaching 100 percent conversion.

At the time of preparing this paper, this new reaction tube equipment could not be operated on account of delay in delivery of substation apparatus which is to furnish power. However, an account of results will be given in a paper in the near future.

In order to successfully put the process into commercial operation it is planned to build reaction tubes adjacent to the crude-oil stills now installed in the various refineries in the country, connect them to the vapor line of the stills, put a perforated gas pipe in the bottom of each still for the admission of natural gas, and after the first benzine "cut" is made, divert the vapors of the heavier fractions through the reaction tubes, which,

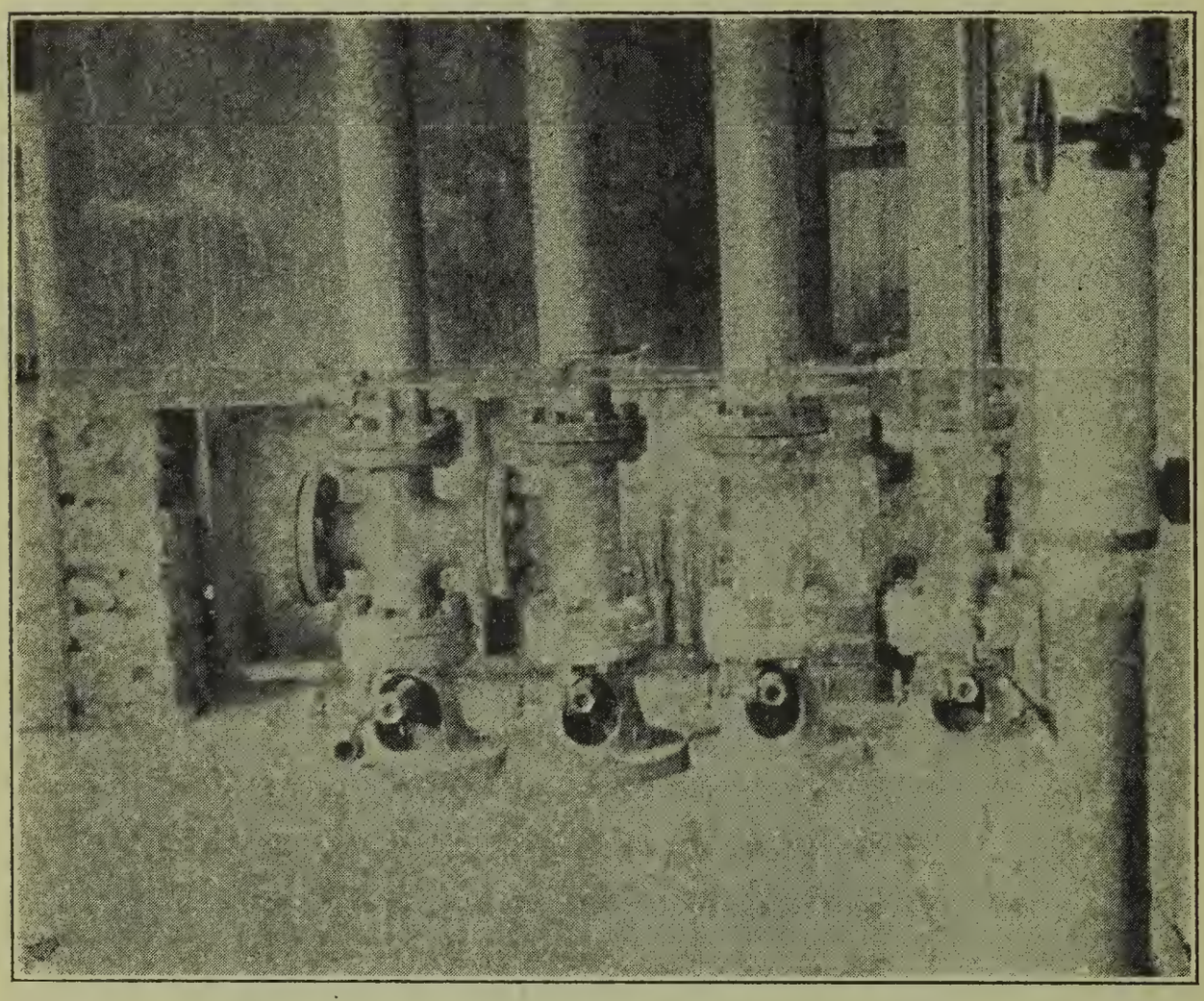

FIG. 12. Lower Part of 34-Foot Reaction Tubes, with Enclosing Coils for Heating.

when mixed with the natural gas which has been admitted by the perforated pipe, may be converted into a crude benzine similar to that which was first taken from the crude oil.

As stated at first, the density of the product may be changed by varying the frequency of the oscillatory current, but by experiment it has been discovered that the product may be more easily changed in density by varying the temperature of the reaction

tubes, and in so doing one would expect that a temperature might be reached which would break down or "crack" the oil vapors with the attendant precipitation of carbon; but so far this trouble has not been experienced, although temperatures have been carried up to 480° C. (about 900° F.). Had this temperature been carried without the presence of the high-frequency current, precipitation of carbon would have been very rapid, and an ill-smelling product consisting largely of the olefines would have been the result.

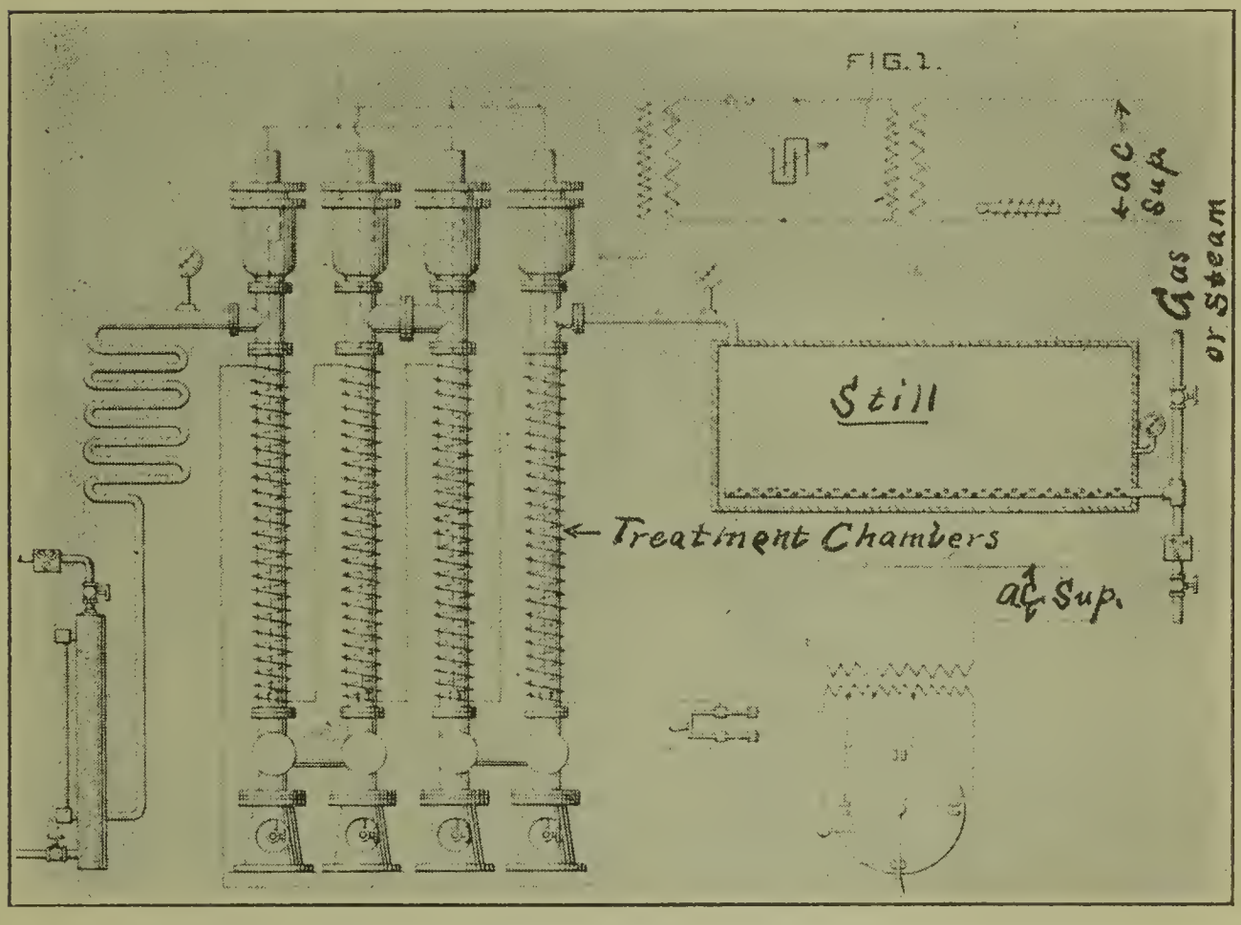

FIG. 1(a). Diagrammatic sketch of electrical connections of oscillatory equipment, heating equipment and control, and method of connecting reaction tubes to still and condenser.

Apparently the ionizing effect of the heated tubes combines with the effect of the high-frequency current, and produces a resultant effect upon the gases and oil vapors, disorganizing, and at the same time reorganizing their molecular formations, this being done without complete separation of their elements.

The possibilities of this process are apparently very great, and it is predicted that the aromatic hydrocarbons, such as benzol and

toluol (which are in so great demand at present for the manufacture of dyes and explosives), will be made by some modification of this process.

Some experimenting has been done in this line, with kerosene (without the addition of natural gas), under a pressure of 80 pounds per square inch (5½ atmospheres), and a temperature

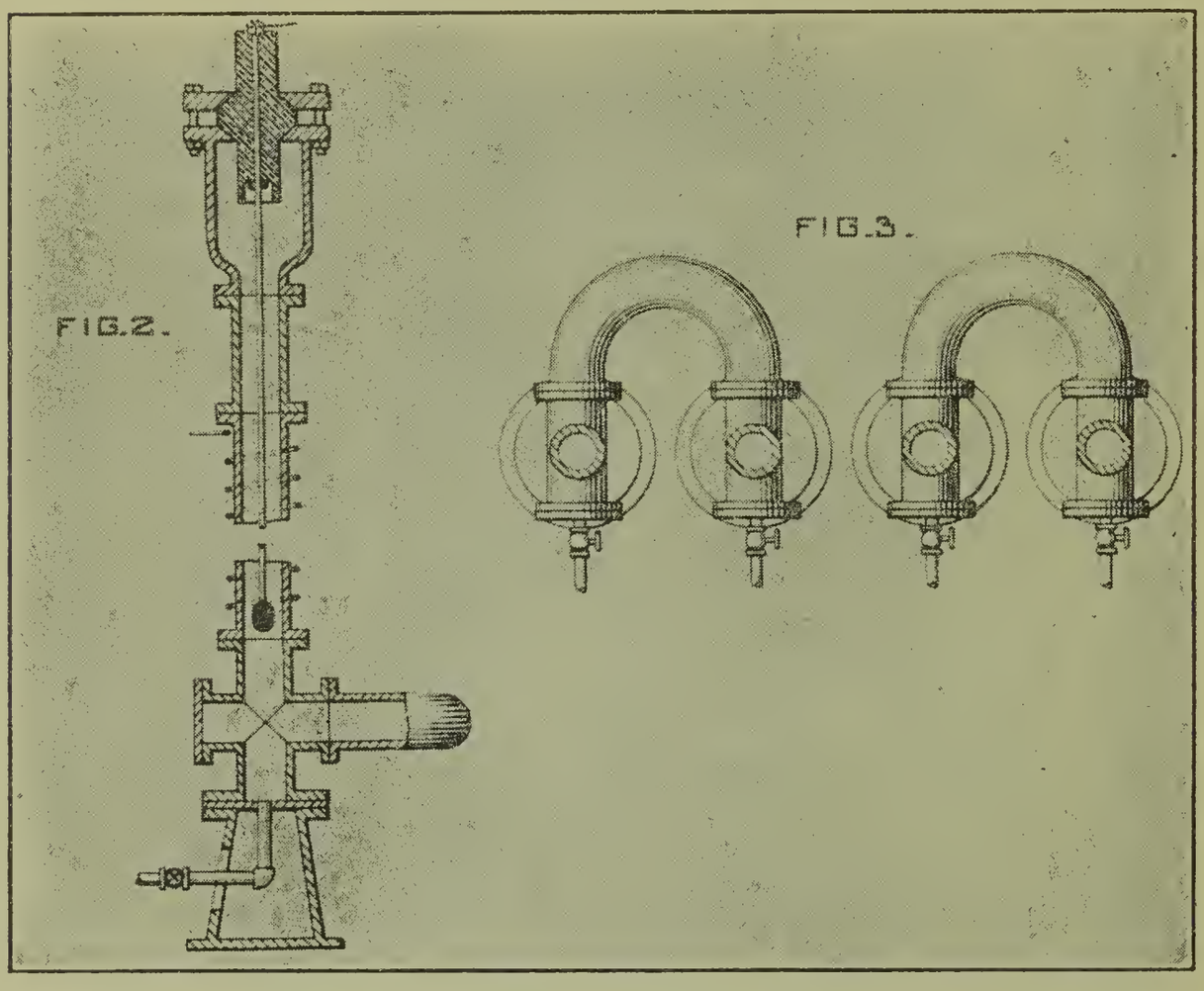

FIG. 2(a). Section of Reaction Tube. FIG. 3(a). Plan of Reaction Tube.

of the treating chambers of 538° C. (1,000° F.), which produced a distillate which seemed to be composed largely of a mixture of benzol and toluol. Complete refining and rectification of these products were not made, but their reaction with different reagents and their ability to dissolve asphaltum indicated a high percentage of these two compounds. Further investigation along this line is contemplated in the near future.

Some of the advantages of this process are as follows: Reac-

tion tube equipment comparatively cheap; easily built and installed; small depreciation, as compared with "cracking" equipment, where precipitation of carbon, together with corrosive action of products of combustion with which they are heated, soon destroys the plates and tubes.

The apparatus is, in reality, an attachment to the ordinary refining equipment, and utilizes the stills and condensers, agitators and tankage already installed.

By converting the distillates between the benzine fraction and that part in which the lubricating oils are found, it is possible to increase the output of gasoline 200 percent.

One kilowatt of electrical energy used in the reaction tubes will convert 1,000 gallons (3,785 liters) of distillate in 24 hours, or a 60-K.W. plant should convert about 60,000 gallons (227,100 liters) per day; a total of 1,440 K.W.-hours are required for the oscillatory current, to treat 60,000 gallons (227,100 liters). It has been found by experiment that 40 K.W.-hours of electrical energy is required for heating the reaction tubes while producing 60 gallons (227 liters) per hour, and if the ratio holds true for a 60,000-gallon (227,100-liter) plant, it would take 39,360 K.W.-hours for heating the reaction tubes, while 60,000 gallons (227,100 liters) were being converted; this added to 1,440 K.W.-hours would make 40,800 K.W.-hours total energy required, which at a cost of 0.6 cent per K.W.-hour (at which price electrical energy can easily be produced at refineries) would make a total of $244.80, or about 0.4 cent per gallon (3.8 liters).

Superheated steam may be used with the gas, or may be used alone. When steam is used, it is decomposed in the reaction tubes, the nascent hydrogen uniting with the hydrocarbons, and the O uniting with C forming CO, which is trapped off at the tail of the condenser, is available for fuel, and may be burned under the still. Artificial gas may be used where natural gas is not available; in fact, any hydrocarbon or hydrogen-carrying gas may be used. Two or more liquid hydrocarbons of different gravities and boiling points may be mixed and treated, and a compound of different characteristics from any of the original compounds obtained.

While operating, the temperature of the oil in the still is maintained a few degrees below the point that would cause it to distill

over, and the gas going in at the bottom of the still rises through the oil and acts as a carrier for the oil vapors. In this way, perfect control of the velocity through the reaction tubes is maintained. Should a porcelain insulator break, or anything happen to interrupt operations, by turning off the gas going into the still, and shutting off the fire, distillation almost immediately ceases.

A commercial demonstration has not yet been made.

DISCUSSION.

L. B. CHERRY: I will exhibit a bottle of gasoline, and make a few remarks. It is with much hesitancy that I have presented a paper on this subject, owing to its novelty, and I therefore expect a great deal of criticism from those present, on account of the conclusions drawn. We have tried in this paper briefly to present a theory, and follow with an account of results from experimentation which seem to support the theory.

The theory is briefly this: If the only difference between our elementary substances was the number of and organization of electrons, and if we could take a hydro-carbon oil in vapor form and mix it with hydrogen or a hydro-carbon gas and impress upon this mixture radio-active energy of the proper periodicity, we would cause the compounds to vibrate together, and after leaving the zone of impressed radio-activity, they would form some natural organization, depending, to a certain extent, on the physical conditions with respect to heat and pressure. In other words, we attempt to thin down the oil with natural gas, to make gasoline.

This paper tells of some of the results of experiments with a small plant, and we expected to have a larger one in operation before coming to the meeting, but the express companies wrecked our transformers, therefore making it impossible to carry out our plans, and give you the results of a commercial demonstration. The transformers are now being repaired, and will soon be in operation. This work has been most interesting. We might with apologies call the product gasoline. It is 52° (Baumé) gravity, 42.75° C. (109° F.) initial B. P., 204.95° C. (400° F.) end point,

and was made from 42° (Bé.) gravity kerosene and natural gas, of which about 78 percent distilled over, under the temperature of 151.55° C. (305° F.). Most refiners use the Fahrenheit thermometer, and Baumé hydrometer. Its low Baumé gravity makes it a superior motor fuel, as you have more weight per unit of volume. I will leave this sample of gasoline for your inspection. See Fig. 13.

H. C. P. Weber[1]: The problem and the method of attack is a very interesting one. I have been looking over the references to a process of similar nature in which condensation products of higher molecular weight have been prepared, yielding materials

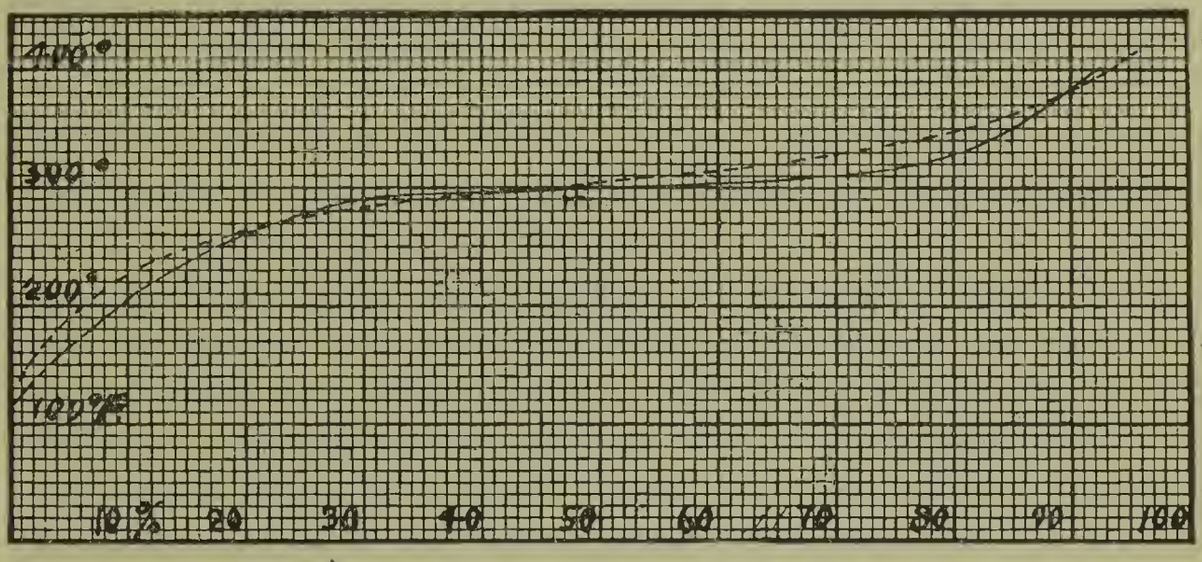

Fig. 13. Dotted Curve: "Straight Run" Gasoline.
Solid Curve: "Cherry Process" Gasoline.

of a waxy nature. The work was done in France or Italy, as far as I recall. I would be glad to know whether Mr. Cherry has similar products in view, in addition to the ones of lower molecular weight.

Further, there should be some relation between the frequency of vibration of the molecule and the most effective frequency of the current. But one would imagine that the molecular vibration frequency would be enormously higher than any available at present. Has any possible numerical or quantitative relation been established between the applied frequencies and those of the compound?

Third, the material obtained being products of lower gravity

[1] Research Laboratory, Westinghouse Elec. & Mfg. Co., East Pittsburgh, Pa.

may be the result of solution of very low molecular weight products, methane for example, in products of comparatively high molecular weight. It would, of course, be difficult to separate methane from heptane, etc., by one or two distillations. I would like to know whether this matter has been carefully followed as a possible explanation of the results.

L. B. CHERRY: First, the matter of treating compounds of higher molecular weight has been taken up by a number of others, particularly by Hemptine, in a process whereby oleic acid ($C_{18}H_{34}O_2$) is changed to stearin ($C_{18}H_{36}O_2$) by the effect of a high-frequency current upon films of the former, brought up on rotating glass plates, in an atmosphere of hydrogen. I have converted the mixed vapors of paraffin wax and natural gas, and raised the mixture nearly to the gasoline point in its gravity and physical characteristics, and we do not doubt that the field of development in the products with heavy molecular weights is very broad.

Second, the frequency we found to be of great importance, and with the apparatus at hand we found that raising the frequency we lowered the boiling point and raised the Baumé gravity. The oscillatory current and the radiant heat of the tubes seem to combine with a resultant effect on the mixed oil vapor and gas, as we were enabled to get the same results, within certain limits, by varying the temperature of the reaction tubes. The molecular vibration periodicity is no doubt very high, but we may strike some lower harmonic and reach that of the molecule sympathetically, just as we strike a low string of the piano and cause the higher ones several octaves above to vibrate. The frequency we employ is extremely high, special apparatus had to be built to accomplish the results, the current from which may be passed through the body without being sensed, and is above the point of audibility, which I believe is about 35,000 vibrations per second. But no numerical relation has as yet been determined between the applied frequencies and those of the compound. We know that the wave lengths of the solar spectrum range from 0.76 micron (red) to 0.38 micron (violet) and that emanations from heated and luminous bodies that come within this range will quickly decompose hydro-carbon compounds, and that extremely high-frequency oscillatory currents produce reactions

between methane and other organic compounds (see Electrochemistry of Organic Compounds, by Löb and Lorenz). Just what the resultant effect is of the combination of radiant heat and high-frequency current, together with a possibility of an electro-magnetic effect by inductively heating the tubes with electricity, just how this causes the reaction between the oil vapor and gas, just what the resultant periodicity is, or whether it is necessary to apply energy of different frequencies to act on the two different compounds—would be interesting to determine.

Third, the suggestion that a solution is possibly formed between the low and the high molecular weight hydro-carbons, such as methane and heptane, does not seem to me satisfactory, as methane being a gas, it seems improbable that enough would stay in solution with heptane to affect very materially its physical properties, unless a molecular reconstruction had taken place.

E. B. Pratt[2] (*Communicated*): From my own limited understanding of the various processes for producing gasoline from a mixture of heavier hydro-carbons and gas, it seems that the process herein described differs from the others in that Mr. Cherry subjects the mixture of heavy hydro-carbons and gas to an electrical discharge of high-tension, high-frequency oscillating current, and thereby gets a large percentage of gasoline in the product. The high pressure and high temperature are used in the other processes also.

Mr. Cherry states that high pressure in his process is not necessary, but gives higher percentages of conversion, and higher gravity. Inasmuch as high pressure is necessary in the other processes where the electrical discharge is not used, it follows that the electrical discharge has the same effect as the high pressure, namely to facilitate the reaction, and when both are used in conjunction, the results are correspondingly better.

The second point to notice is the small percentage of hydrogen necessary to convert the heavy hydro-carbons to gasoline. And he states that on account of the cost of producing hydrogen, he prefers to use natural gas. This deserves both commercial and chemical consideration. The commercial aspect is that hydrogen may be purchased in tanks from any one of the many firms (scattered throughout the country from the Atlantic to the Pacific)

[2] Mutual Benefit Life Insurance Co., Cleveland.

which produce hydrogen and oxygen by the electrolysis of water. Therefore, if a proposed plant for synthetic gasoline were to be located where natural gas was not indigenous, hydrogen would be the more universal material. The chemical aspect is that it is hydrogen which the process demands, and not methane. The latter is used solely because it is a source of obtaining hydrogen, and cheap. Pure hydrogen instead of methane would mean a less complicated chemical action within the retort, and, after experimental adjustments, probably would give a larger percentage of gasoline.

Also worthy of note is that Mr. Cherry says that the density of the product may be more easily changed by variation in temperature than by variation of frequency of current. Therefore temperature seems to be of more importance than either the electric current or pressure. This strikes me as being very important. In the other processes, if my information is correct, carbon is precipitated at the higher temperatures, whereas, Mr. Cherry states, no carbon was precipitated at 480° when the electric current was used. He goes on to state that when a still higher temperature was used (538° C.) a distillate was produced which seemed to be composed largely of a mixture of benzol and toluol. Now, if Mr. Cherry can add to the table given on page 346 the temperatures at which these various hydro-carbons are completely decomposed into carbon and hydrogen gas, it seems to me that he has the basis of almost any kind of synthetic composition desired, provided he uses pure hydrogen instead of methane.

If Mr. Cherry will send me the temperatures of volatilization and decomposition of these hydro-carbons, or of those which he uses, I may be able to give him some helpful information, as I have had some experience with hydro-carbons at high temperatures in connection with another process not in any way related to this one.

L. B. CHERRY: Mr. Pratt's remarks concern the operation of the process under high pressure and temperature as compared to performance at atmospheric pressure. It seems reasonable to conclude that the effect of pressure and temperature on the compounds under treatment is to vary their molecular vibration periodicity, and the higher percentage obtained under pressure mentioned in my paper was obtained when my equipment had a

three-foot length of reaction tube, and an oscillatory generator that was insufficient in the generation of high enough voltage and frequency. Since then we have built a new plant, increased the length of reaction tubes to ten feet, and secured 78 percent conversion at atmospheric pressure. A further increase in length to 34 feet secured a conversion of 84 percent, and former experiments cause us to believe that by running the remaining 16 percent through the equipment we can convert 84 percent of that, which causes us to conclude that the relative time that the compounds are under treatment determines the percentage of reaction which may be expected, and the combined action of heat and high-frequency current determines the quality of the product

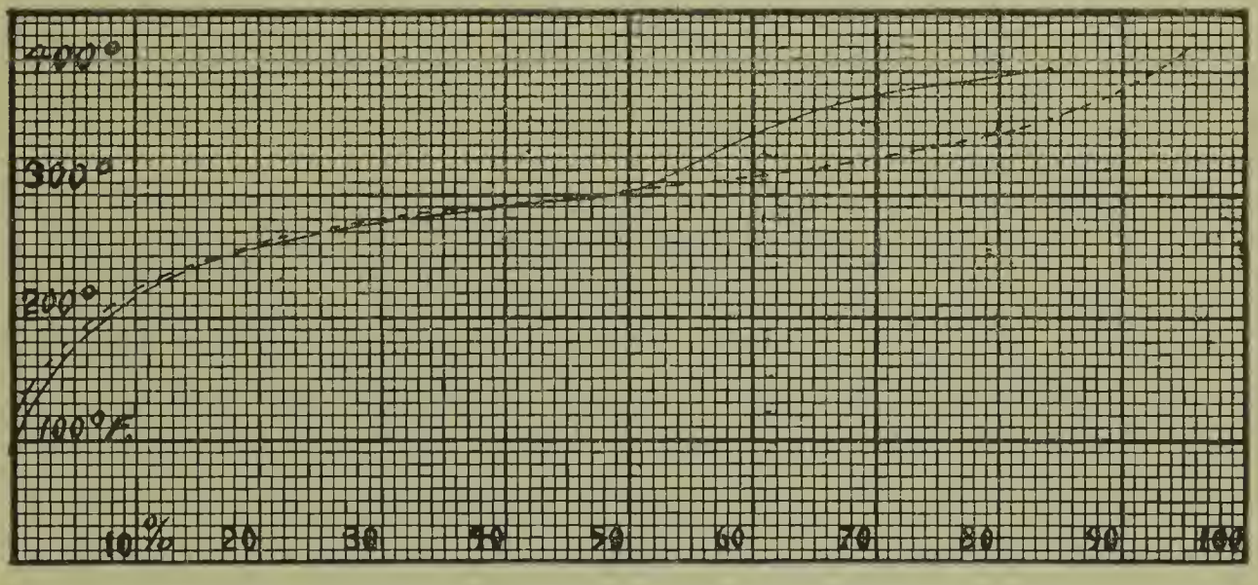

FIG. 14. Dotted Curve: "Straight Run" Gasoline.
Solid Curve: "Cherry Process" Gasoline.

obtained. During our run of the plant the latter part of October, 1917, we had the 34-foot reaction tubes connected to a seven-barrel still, which distilled the kerosene over at the rate of about one gallon a minute, with which apparatus we secured (See Fig. 14) a conversion of 84 percent of the kerosene into light compounds with an end B. P. of 400° F., and while so doing we forced into the still 2 cubic feet per minute of natural gas at a pressure (when metered) of 2 pounds per square inch. The increase of length of reaction tubes from 10 feet to 34 feet should have given us a proportionate increase in conversion; as it was, the increase was from 78 percent to 84 percent. However, we account for this apparent discrepancy by the fact that more kero-

sene was being distilled per minute when using the longer reaction tubes (which were of same diameter) than was distilled when the tubes were 10 feet long, and the gases had a comparatively higher velocity, which would comparatively decrease the time they were under treatment, per unit of length.

I am sorry that I cannot give the temperatures at which complete decomposition takes place, as suggested by Mr. Pratt, as there are so many conditions, such as shape of the still, material of which it was made (the catalytic action has to be accounted for), etc., all of which would make it difficult to get reliable data. Then I know of no method that would *completely* decompose a hydro-carbon into its elements by the application of heat, as there would be a polymerized lot of compounds that are sure to form, judging from what little experience I have had. However, with the top of the still well insulated to prevent cracking, there is very little decomposition under 600° F. for kerosene and 700° F. for gas distillate.

W. J. Van Sicklen[3] (*Communicated*): In this paper Mr. Cherry presents an interesting electrochemical method for the production of gasoline from hydro-carbons of higher boiling points. By passing suitable mixtures of natural gas and vapors from heavy oils through a zone in which electrical discharge of high tension and high frequency is maintained he finds that a considerable percentage of the original oil is converted to gasoline. He points out that this transformation of the heavier oil is brought about by the electrostatic and electrochemical conditions set up in the treatment chambers, and that a high conversion yield is possible under conditions attainable in practice.

In view of the present status and future plans for the process, as set forth by Mr. Cherry, I should like to ask about the recovery yields of the various products that should be obtained in the proposed extension to commercial production, assuming that natural gas is available. Mr. Cherry states that by using a ten-foot reaction tube a 78 percent conversion of kerosene to gasoline was obtained, but does not mention whether this represents the percentage of kerosene plus gas converted to gasoline or some other value. It would be interesting to know also if he has determined the percentage of original materials converted

[3] Chemist, San Francisco, Cal.

to gas and to carbon, since a considerable percentage of these products might result owing to the high temperature employed in the treatment chambers.

In discussing the relative influence of frequency of the oscillatory current utilized and temperature of the reaction tubes in lowering the density of the oil products obtained from kerosene, the author mentions that the latter appears more effective but that the former is necessary if cracking and attendant precipitation is to be prevented. He also mentions that by thermal cracking alone a much larger percentage of olefines and other unsaturated products would result. It would be interesting to know what specific influence the oscillatory current exercises in the production of unsaturated hydro-carbons irrespective of the thermal treatment. It is possible that Mr. Cherry has determined the percentages of original materials converted to saturated and unsaturated oils from an analysis of the products obtained both with and without the utilization of the oscillatory discharge, while keeping the thermal and other conditions constant.

I would like to ask if the author has ever tried to utilize the gaseous products obtained in the process of heating and thus maintaining the reaction tubes at the proper temperatures, instead of using the more costly electrical energy for that purpose. If this were done would it not make the process more economical as regards production costs, or has the induction heating as a result of the eddy currents developed in the walls of the reaction tubes some specific action in transforming the heavy hydro-carbons to those of lower boiling points?

L. B. Cherry: The fact that we had no appreciable amount of uncondensable gases and carbon precipitaton is remarkable, and leads us to expect a 100 percent conversion, on a volume basis. That is, we believe the losses will not exceed the increase in volume, due to raise in gravity, and that we will be able ultimately to secure as many gallons of marketable gasoline as we had of distillate to start with. Reference in my paper to different percentages of conversion mean that a sample of the synthetic benzene was caught at the tail of the condenser during the run, fractionated by distillation, and the percentage "over" under 400° F. represented the percentage of conversion. The temperature

carried on the reaction tubes is sufficient to crack the oil vapor, were it not for the oscillatory current, as was evidenced by cutting off the high-frequency current during the run, for a few minutes, and when it was turned on again the free carbon which had been precipitated during that time and was sticking to the inner walls of the tubes, was loosened up on continuing the high-frequency current, and coming over with the distillate would color it as black as tar, but in a few minutes all free carbon was eliminated and the distillate would clear up and continue to run clear.

There seems to be a decided advantage of the use of electrically heated tubes, on account of the more sensitive control of the heat. It was noted that a variation of 20° F. made a great change in the product. The cost of so doing is not prohibitive. By actual watt-meter reading, the consumption of electrical energy for both the heating and the oscillatory current was 628 watt hr. per gallon of kerosene treated, which at the prices quoted from large central stations is about one-half cent per gallon. The unused gases can be put back through the still and through the reaction chambers, where they ultimately are mostly converted into aromatics; or, the gases unconsumed may be burned under the still, but the use of fuel for heating the reaction tubes, with the attendant depreciation of furnace and tubes, and difficult control of the temperature, I believe would show a lower commercial efficiency than where the tubes are electrically heated.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

CHLORINE AS A WAR FACTOR

By Wm. D. Marshall*

[Abstract.]

A review of the uses of chlorine in war service, particularly of recent practice in sterilizing drinking water in the field by use of liquid chlorine, and of the production of hypochlorine solution with 0.45 to 0.50 percent available chlorine, for field hospital use, by using carefully weighed ampoules of liquid chlorine with a fixed amount of water and soda, for the Carrel and Dakin treatment.

During the first year of the war, reports from abroad have frequently reached this side to the effect that chlorine was used in the form of gas attacks, releasing it from cylinders or containers against the position of the enemy.

My own experience with chlorine, as well as what I have heard from others on the subject, leads me to believe that it alone could never have caused such agony and internal injuries as are described in the various articles, because in my practice I have never seen nor heard of anybody permanently injured by inhaling chlorine, although the temporary inconvenience and pain caused by it seemed at times rather serious. This of course refers to conditions such as prevail in factories, to which conditions on the battlefield must be almost identical or at least very similar. That is, that a person is subjected to fumes of great dilution for a limited space of time, for periods lasting from several minutes to even several hours, according to the concentration of the gas in the air, and afterwards gets out again into the fresh air.

* With Electro Bleaching Gas Co., New York City.

Whatever bad reputation chlorine has incurred as a poison gas, deservedly or undeservedly, it is more than making up for it by the beneficial applications to which it has lent itself for war purposes in sanitation, sterilization of wounds, etc.

The first revolutionary progress in the application of chlorine for water sterilization was not made until the latter part of 1912, when liquid chlorine was first introduced to the sterilization of city water supplies, through accurate measuring and regulating devices which permitted its application in absolutely quantitative amounts in exact proportion to the amount of water to be treated. This method was developed at such a pace, that today, less than five years since its inception, well over one thousand cities and towns in the United States and Canada are employing this method and the typhoid charts of the Board of Health of various communities prove conclusively the rapid drop of typhoid fever cases immediately after the introduction of chlorine sterilization.

A method such as the above, it is evident, is applicable mainly to stationary water works, where a steadily flowing body of water has to be treated. The sterilizing apparatus itself, however, need not be stationary owing to its small size, lightness and compactness and as a matter of fact a special type of small and self-contained, so-called emergency apparatus has been devised, which can be shipped or carried by one man, from place to place, wherever urgent necessity requires immediate sterilization, but even in this case the water works where the apparatus has to be put in use is of a stationary kind.

In war, stationary conditions prevail only in the minority of cases, for instance in permanent camps, etc., and for these, particularly if of fairly large size, where considerable quantities of water, either flowing or collected in large reservoirs or tanks, are to be sterilized, the same type of apparatus may be employed to advantage. For smaller units, however, and such of a more temporary character, where quantities of one or several barrels will suffice, other methods have to be resorted to. Tablets and ampoules containing mixtures of bleaching powder and sodium carbonate or sulphate, in known quantities, have been recommended, but do not seem to meet with much success, the lack of

which is caused undoubtedly by their insufficient stability and—as a consequence—the uncertainty as to sterilizing efficiency, precipitation of lime sludge, etc.

As a result of a large number of experiments along these lines we feel convinced that in preference to any of the above an ampoule filled with chlorine water of medium concentration furnishes the best portable form of chlorine as a sterilizing agent for relatively small quantities of water.

Chlorine water, although of very unstable character when made under commercial conditions where impurities capable of catalytic influence, exposure to light, escape of chlorine into the air, etc., are possible, yet if made under conditions of the utmost cleanliness and elimination of all of the above factors for premature decomposition, is a very stable compound, much more so than bleaching powder and most of its equivalents.

These ampoules are at present made in two sizes—one containing sufficient chlorine to sterilize one fifty-gallon barrel of water, and the other smaller one for canteen size, or approximately one quart.

An additional advantage over the use of hypochlorites is that nothing but chlorine gas and water enters the water to be sterilized. All lime, soda, salts, etc., are absent. The canteen size is of a form and so small that it can easily be carried by a man in his cigarette case and is always ready for instantaneous use without any necessity of waiting for dissolving, settling, etc. An important factor in this respect is that chlorine water, when made and sealed into tubes under proper precautions, is less liable to decomposition through body temperature than bleaching powder.

Another large use to which chlorine has been applied in the present war is the sterilization of infected wounds, for which purpose Dr. Carrel of the Rockefeller Institute has worked out a very ingenious method of keeping the entire surface of the wound saturated with the solution.

The solution as prepared first by Dr. Dakin, working in conjunction with Dr. Carrel, contains 0.45 to 0.50 percent available chlorine, and small amounts of sodium carbonate and bi-carbonate,

but no free caustic, and was made by dissolving certain amounts of bleaching powder in water, precipitating the lime with a definite quantity of sodium carbonate and bi-carbonate, allowing it to settle and filtering the settled solution.

Apart from the inconvenience and amount of time (12 to 24 hours) required for the settling and filtering, the necessity of testing for available chlorine before and after the addition of soda caused great inconvenience. In order to be effective, 0.45 to 0.50 percent of available chlorine, as mentioned above, must be present. If below 0.45, it is lacking in efficiency, if above 0.50, it is irritating to the surface of the wound.

Recognizing these drawbacks, we have recently developed a method by which, instantaneously, a clear solution of absolutely definite strength can be obtained. The method consists in sealing exactly five grams of liquid chlorine in glass ampoules, which represent tubes of about 8 to 9 mm. outside diameter, ½ to ¾ mm. wall, 20 to 23 cm. long.

These ampoules before leaving the laboratory are tested at a temperature of 75° C., which raises the pressure of liquid chlorine, which at ordinary temperatures is about 80 to 90 pounds per square inch, to over 350 pounds. In view of this immense pressure, the slight wall thickness of the glass ampoules necessary is remarkable.

In order to prepare the solution, one liter of water is poured into a two and one-half liter, so-called acid bottle. A tube containing an amount of soda sufficient to absorb the five grams of chlorine together with the necessary excess of alkali, as required by Dr. Carrel's directions, is emptied into the water and dissolved by shaking. The liquid chlorine ampoule is suspended pendulum-like with its butt end fastened in a short piece of rubber tubing attached to the lower end of the rubber stopper closing the bottle. The bottle is now covered with a canvas bag and the ampoule broken inside of the bottle in contact with the alkali solution by violent shaking. Shaking is continued for a few more seconds to make sure that all the chlorine is absorbed and the solution is then ready for immediate application. Frequent tests of its strength have never varied more than from 0.47 to 0.50 percent available chlorine, and it is therefore well within the requirements.

The method of suspending the ampoule in the bottle causes almost instantaneous breakage of its tip, without any detriment to the bottle itself. Before we used the attachment for suspending the ampoule, we broke quite a few bottles, not by the pressure of the chlorine suddenly liberated, which is amply taken care of by the air space provided, but the recoil of the ampoule, which is now taken up by the rubber stopper, was of such force as to punch a hole clear through the wall of the bottle as round and clean as a bullet hole.

For large field hospitals, in which from one to five fifty-gallon barrels of this hypochlorite solution are used daily, the ampoule method, though entirely satisfactory, would no doubt be too expensive in the long run.

To meet this novel situation (fortunately possible only under the present war conditions) a non-returnable metal package has been devised, containing 945 grams of liquid chlorine, which, together with a package containing the corresponding amount of alkali, need only to be dissolved completely in fifty gallons of water to yield at once the finished solution of standard strength.

The above figures of one to five barrels, or 190 to 850 liters, of hypochlorite solution per day per hospital may, though insufficiently, convey a faint idea of the awful sufferings of mankind on the other side, particularly if one is simultaneously conscious of the fact that the solution is applied to infected wounds only, and that not more than 120 to 250 c.c. is required per wound per 24 hours.

Only in the light of such figures and the records of results accomplished can the revolutionizing influence of chlorine sterilization be properly appreciated.

According to several authors mentioned in Wm. O'Neill Sherman's article on this subject, in "Surgery, Gynecology and Obstetrics," 80 percent of amputations are due to infections, and 75 percent of the deaths occurring after the first 24 hours are due to the same cause, whereas Dr. Carrel's results with chlorine as sterilizing agent in the hospital at Compiegne show a record of 99 percent of the infected wounds healed.

DISCUSSION.

PRESIDENT C. G. FINK[1]: The Carrel and Dakin treatment, referred to in Mr. Marshall's paper, is working wonders for our Red Cross mission. To be able to transport chlorine in liquid form, and to produce a strong sterilizing solution in the simple manner here described, is a valuable step forward. This new method of wound sterilization, as outlined by Mr. Marshall, is a great improvement over the old.

W. B. SCHULTE[2]: Another practical way of carrying chlorine for sterilizing is in combination as sodium chloride, from which it can be liberated by electrolysis. A small and practical device to accomplish this consists of two graphite electrodes 2 cm. in diameter, on conducting rods connected to a socket, and a 4.5-volt pocket flash-light battery. Between the electrodes is a round disc of filter paper which has been impregnated with the sodium chloride. The entire outfit of battery and sterilizer weighs only six ounces.

When the electrodes are immersed in a cup of water, a strong solution of sodium chloride is formed between them in the paper and electrolysis takes place with the formation of the usual products, including chlorine and sodium hypochlorite. The action is indicated by bubbles which immediately arise from the electrodes, and after a few seconds the characteristic odor of chlorine is noticeable. No salt taste is imparted to the water, as the amount which goes into solution is so small; the chlorine odor, which is not always disagreeable, disappears after a short time.

It takes only a few seconds to liberate enough chlorine to sterilize a cup of water, but it is usual to sterilize for a minute, during which time the electrodes are used as a stirrer to mix the chlorine into the water. A small tubular flash-light battery has enough energy to sterilize from 500 to 1,500 liters of water under these conditions—enough to supply a man for several months.

The device is not dependent on the use of a flash-light battery,

[1] Director of Research, Chile Exploration Co., New York City.
[2] Burgess Laboratories, Madison, Wis.

as a small hand-operated generator weighing 1¼ pounds will also provide the proper energy.

The concentration of chlorine of course is small, and the sterilized solution is not strong enough to disinfect wounds for the Carrel and Dakin treatment, but it has been demonstrated that the device sterilizes drinking water quickly. Bacteriologists who have experimented with it are astonished at the rapidity of action and we attribute it to the activity of the chlorine in the nascent condition.

A summary of some of the tests conducted with the device are as follows:

1. 200 cc. of inland lake water was treated. One cc. of water was removed every half minute and tested for bacteria. The untreated water contained 325 bacteria per cc. After one-half minute sterilization this was reduced to five. The five bacteria which were left were the harmless spore formers, which are not attacked by chlorine.

2. A fresh typhoid culture was added to 200 cc. of water, bringing the count up to 1¼ million per cc., which is an extremely strong concentration. After two minutes electrolysis the water was practically sterile. The bacteria which were left were not typhoid.

3. To lake water containing 2,000 bacteria per cc. was added 600,000 typhoid per cc. After four minutes sterilization four bacteria were left. A microscopic examination showed these to be higher bacteria and spore formers. No typhoid was present.

W. D. Marshall (*Communicated*): The small portable apparatus for sterilizing drinking water, as described by W. B. Schulte, seems to be indeed very handy and ingenious, and the weight of only six ounces for the entire outfit is very small.

However, despite these advantages, I am uncertain as to whether a soldier in the field would prefer an electrolytic outfit of this type to a glass ampoule, as described above, containing a measured amount of chlorine water sufficient in quantity for instantaneous sterilization.

It seems to be the general tendency to give preference to mate-

rials marketed in definitely measured unit quantities. This tendency of the public in general, as well as of professional men, such as physicians, etc., can be observed daily, and while an electrolytic cell of large type could perhaps be constructed for large disinfecting units, yet I doubt if it could overcome the competition of tablets or ampoules for the requirements of the individual soldier.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

THE APPLICATION OF RADIUM IN WARFARE

By Charles H. Viol and Glenn D. Kammer.*

[Abstract.]

The sources of radium are described, and its extraction briefly noted. The use of radium tubes in therapy is alluded to. The making of luminous paint, consisting of crystalline hexagonal zinc sulphide with from 0.025 to 0.30 milligram of radium per gram of sulphide, is described in some detail, and the factors which control its luminosity and its active life are discussed at length. Various applications of this paint are described, and the best kind of composition for various purposes discussed.

While the production of radium is not carried out by electrochemical processes, the action of this wonderful material is so intimately bound up with electrical effects that it is not inappropriate to consider the applications of radium in warfare in this symposium.

It was in Pittsburgh in 1913 that a pure radium salt was first prepared in America in the Research Laboratory of the Standard Chemical Company, and since then Pittsburgh has fairly earned the distinction of being the world's greatest radium-producing center. In the midst of industries whose output is measured in thousands or millions of tons, an industry whose total output in nearly five years amounts to about one ounce is likely to seem small, yet this production of radium by the Standard Chemical Company, of Pittsburgh, is the most notable of any in the world, and the output represents more than a fourth of the estimated total world's stock of high purity radium. This radium is produced from carnotite, a uranyl vanadate, mined in southwestern Colorado and southeastern Utah.

* Research Chemists, Standard Chemical Company, Pittsburgh.

From three hundred to five hundred tons of the available low grade carnotite must be treated to yield one gram of radium element in the form of a high-grade salt. The raw ore is first concentrated mechanically, this being possible because of the makeup of the ore, which consists mainly of a sandstone impregnated or cemented by the carnotite material. The concentrates carry approximately 10 milligrams of radium per ton, and, after removal of the vanadium and uranium, are treated to yield all the barium in the ore, this barium carrying with it the radium. Radium barium chloride containing about one part of radium per 500,000 parts is then subjected to a process of fractional crystallization which ultimately gives a nearly pure radium salt, together with barium salt tailings which contain less than one two-hundredth of the original radium.

Radium has found an important place in therapy and in this field it may be considered as a war material, since it is the most valuable agent known for the treatment of scars and keloids, resulting from wounds and burns, and certainly war increases greatly the number who require treatment for such conditions. Radium tubes applied in chronic infected wounds cause remarkable effects in hastening the healing of these often most intractable conditions. For those interested in this phase of the use of radium I will cite the papers of Dr. W. H. Cameron in the Pennsylvania Medical Journal, Vol. XIX, 449-53, March 1916, and Dr. J. Barcat, in Le Progrès Medical, May 20, 1916, where the subject is well summarized.

A use of radium which has a more direct application in warfare, and which now is becoming of almost as great importance as the medical use, is in the production of the so-called permanently luminous compounds. This luminous material finds a wide range of uses in all forms of dials and indicators that must be seen in darkness or semi-darkness. One most important use is on the instruments of aeroplanes, for night flying. The intensity of the luminescence of this compound is such that the accommodation of the eyes is not impaired, as would be the case if a brilliant electric light were used, and so observations of the surroundings can be made more readily. With a luminous dialed instrument there is not the danger that a defective battery or generator or a

broken wire or bulb would entail, the luminous-dialed instruments being useful until the instrument itself is out of commission.

The production of a suitable luminous compound, the methods of applying this compound, and the methods used to check the luminosity of the compound before and after application, have all presented problems of some difficulty, and since much remains to be done in this field, a brief discussion of these points is not without interest.

Phosphorescent compounds are those which when exposed to suitable light are capable of storing up some of the energy, which is subsequently emitted in the form of light. A number of phosphorescent substances are known in which the luminescence persists for some time. Of these, the best known was the specially prepared calcium sulphide, which formed the base of Balmain's patented luminous paint. However, exposure to light is absolutely essential to the production of any degree of luminescence, and after such exposure this material emits a bluish light for only several hours. It was first used about 1877 on watch and clock dials, etc., but was practically discarded about twenty years ago, largely because of the necessity of first exposing the articles to light to render the luminous paint effective.

Early in this century, after the discovery of radium and other strongly radioactive substances, such as actinium, it was found that the rays of the radioactive substances were capable of exciting certain materials to the emission of visible light, even when not previously exposed to light—the necessary energy being supplied from the radioactive material. Of all known materials phosphorescent zinc sulphide, so-called "Sidot's hexagonal blende," gives the greatest luminescence when exposed to the rays of radium, most of the light from the zinc sulphide being due to the numerous scintillations produced by the bombarding of the zinc sulphide crystals by the *alpha* rays.

Phosphorescent zinc sulphide consists of a specially prepared crystalline form of zinc sulphide which, when mixed with an amount of radium, continues to emit a greenish yellow light, the intensity of the luminescence being dependent on the quality of the zinc sulphide and the proportion of radium used.

It has been known that many phosphorescent substances respond strongly to the action of ultra-violet rays and X-rays; however,

the substances which are very luminescent under the action of these rays (such for example as Balmain's calcium sulphide, and native willemite), do not necessarily produce the brightest luminous mixtures when radium is added. To test the suitability of any given material, it is either necessary to add radium directly to the substance and compare the resulting luminosity with that of a good grade of zinc sulphide containing an equivalent amount of radium, or otherwise to bring *alpha* rays to play on the materials. We have found a polonium plate about 20 millimeters in diameter most useful, since this preparation emits an intense *alpha* radiation. With a suitable polonium plate a superficial luminescence can be obtained in zinc sulphide equal to that obtained by the addition of several hundred micrograms of radium per gram of zinc sulphide. By holding this plate over two adjacent specimens of zinc sulphide or other material, it is very easy to determine the relative values of their *alpha* ray luminescence.

For five years we have sought in the Research Laboratory of the Standard Chemical Company to improve the quality of zinc sulphide, so that a given amount of radium per gram of zinc sulphide would yield the maximum luminescence. In this quest we feel that we have been successful, and so far we have not found material made elsewhere which equals our zinc sulphide.

Naturally we cannot go into the details of the making of this zinc sulphide, but suffice it to say that in addition to fulfilling the requirements with regard to luminosity, the material also is satisfactory as regards the fineness of the powder (important in the application of the compound), and, because of its high luminosity, the luminous compound has as long an effective life as is to be obtained with zinc sulphide. Our "Luma" containing 215 micrograms of radium per gram now shows, for example, a luminosity 2.4 times that required by the National Physical Laboratory to pass the Admiralty Standard, which requires the use of the above proportion of radium.

The luminescence of the zinc sulphide is roughly proportional to the radium content, falling off somewhat as increasingly large amounts of radium are added. Commercially luminous compound is in use containing from 25 to 300 micrograms (millionths of a gram) of radium element per gram of the mixture. Careful photometric tests have demonstrated that the rate of decay of the

luminescence is proportional to the amount of radium, so that compounds which initially are brightest show the most rapid falling off in luminescence, while compounds containing proportionally less radium are not initially so brilliant, but have a longer effective life.

The decay in the luminescence of compounds in which radium is the exciting agent is not due to change in the radioactive substances, since it is well known that radium changes so slowly as to require 1,700 years for its half decay—approximately 1/25th of 1 percent change taking place in one year. The change in the zinc sulphide, however, is not so slow, which accounts for the diminishing luminescence of the compound with time. There are many points in connection with light emission from zinc sulphide that still remain to be explained. However, it may be said that the behavior of the substance indicates a capacity for sending out only a certain total amount of light, so that where a greater luminosity is excited by the use of a larger proportion of radium, the life of the zinc sulphide is proportionately shortened.

It is evident, then, that there are two points of great importance to be considered by the purchaser of luminous compounds—the initial luminosity as determined by a photometric test, and the quantity of radium or other radioactive substance used per gram of the compound. These two points must be known, for the luminosity of the material alone cannot be regarded as a criterion of its quality, since it is possible to produce a suitably high luminosity in an inferior quality of zinc sulphide by the use of a relatively large amount of a cheaper radium substitute—mesothorium or radiothorium—the commercial supply of which, so far as we are aware, is all of German origin. While the price and initial luminosity of such material might compare favorably with that of a compound made with a superior zinc sulphide containing radium, it is evident that the life of the inferior product would be proportionally shorter, because of the increased amount of radioactive substances used to give the necessary luminosity. Since the price of the luminous compound is dependent largely on the radium content, it is only reasonable that the purchaser shall be advised of the radium content. The manufacturers who are using mesothorium and radiothorium are not able to say definitely how much of these products they use, because of the analytical diffi-

culties in the quantitative determination of these products and the comparatively rapid rates of decay of mesothorium and radiothorium.

In the application of the luminous compound for its various uses it is mixed with a suitable vehicle, usually some form of clear transparent varnish, the resulting thick paint being applied by means of a fine stiff-haired brush or a fine stylus. The difficulties to be overcome in this connection are dependent on the nature of the material and surface to be painted, as well as the nature of the surroundings. Paper, porcelain, mica, and metal dials of all sorts bearing figures and lines are now used. In the liquid-damped compasses, such as the aero-compasses, the card floats in alcohol, thus introducing a further requirement that the luminous paint resist the action of this solvent. The varnish most suitable for this purpose is yet to be found. For some instruments, such as aeroplane instruments, the requirements of luminosity are of most importance and outweigh the other considerations of cost and length of effective life. For such purposes the grades of luminous compound are prepared which give the highest initial luminosity, due to a larger proportion of radium, and having, consequently, a shorter effective life. For many other purposes, such as the use on marching compasses, wrist watches, etc., luminous compound of lower initial luminosity is recommended, and this material has the advantage of lower cost and longer life.

The methods for the exact photometric measurement of the luminosity of these compounds have not yet been worked out so as to be entirely satisfactory, by reason of the inherent difficulties in matching faint luminosities, particularly where the standard varies somewhat in color from the light emitted from the compound. A great deal of photometric work of this sort has been done at the National Physical Laboratory, Teddington, England. In December, 1915, the British Admiralty requirement for luminous compounds for use on aeroplane compasses and indicating dials set as the standard (100 percent) the luminosity of the best grade of phosphorescent zinc sulphide activated by the addition of 0.40 milligrams of pure crystalline radium bromide (equivalent to 215 micrograms of radium element) per gram of the compound.

In the June 15, 1917, number of the Proceedings of the Physical Society of London, Patterson, Walsh and Higgins contribute an

extensive paper on "An Investigation on Radium Luminous Compounds" which they have been carrying on at the National Physical Laboratory in answer to a request by the British Admiralty for information and assistance in connection with the use of the compound for dials of instruments used in the Navy. Since our interest has been in the production of the compound rather than quantitative determination of its luminosity, we would refer any one interested to the paper cited for the best information now available on the subjects of the measurements of the luminosity and radium content of radium luminous compounds, the measurement of the luminosity of the markings of luminous dials, the best dimensions for luminous markings to secure maximum legibility, data regarding the decay of luminous compound, both in the dry powder form and after application to dials, and considerations governing the choice and specification of radium luminous compound.

With regard to the decay of luminosity of the material, it is found that the luminosity of the mixture increases for the first two to three weeks after the radium is mixed with the zinc sulphide, when a solution of radium salt is used. Where a dry radium salt is used the maximum is attained sooner, since there is less loss of emanation by this method. For a grade of compound containing 215 micrograms of radium per gram of mixture, the National Physical Laboratory measurements show for the dry powder an almost logarithmic rate of decay from the maximum for about 200 days, after which the rate of decay becomes smaller than required by an exponential law. After about 500 days the luminosity seems to reach a value that is sensibly constant, according to Patterson, Walsh and Higgins, although these authors state that further measurements will be required to settle this point definitely. For the compound mentioned above, the luminosity fell to 50 percent of the maximum luminosity in about 130 days after the preparation of the mixture, and by 500 days the luminosity had fallen to about 22 percent of the maximum.

When the luminosity of the same grade of compound (215 micrograms per gram of mixture) is determined after application, the maximum luminosity is found to be from ⅓ to ¼ of the maximum luminosity of the dry powder, and the effective life is

longer, requiring about a year for the half decay as compared with 130 days for the dry powder.

The measurements on the decay of luminosity of dry powders of different radium content show that those with less radium per gram of mixture decay more slowly, and the rate of decay of luminosity in the applied material has been found to correspond roughly with the decay rate for a dry powder having the corresponding luminosity.

For watches and push buttons, etc., of a better grade, an effective life of ten years or more is desirable, and assuming that a luminosity of 25 percent of the original is still satisfactory, it can be said that a compound containing between 50 and 100 micrograms of radium element per gram of mixture should answer this requirment. Compounds with more radium than this would have a shorter effective life, and compounds with a smaller proportion of radium, a longer effective life.

Radium Research Laboratory,
Standard Chemical Company,
Pittsburgh, Penna.
August, 1917.

DISCUSSION.

GEORGE F. KUNZ[1] (*Communicated*): To prevent a misconception, I wish to state (although I regret exceedingly to do so) that I note with some surprise that this paper fails to make any reference to the early workers in this field. Apparently the authors were ignorant of, or purposely ignored, the early work done by me, in evidence of which there are patents issued by the United States Government, the first in 1903, on things which they bring forward as "new."

I applied radium mixtures to the first watch that ever had markers and hands coated with a radium compound, and also made many other like objects in reference to which my patents may be referred to; and have also been instrumental in drawing attention to many of those who have used this method.

[1] Tiffany & Co., New York City.

PRESIDENT C. G. FINK[2]: Among the applications of this luminous compound, one is very interesting, and that is the illumination of gun sights. A number of big guns have been thus equipped. Mr. Miner, has radio-active thorium been used for these gun sights?

H. S. MINER[3]: Not to my knowledge. Mesothorium has been used in that connection.

N. E. DORSEY[4]: As stated by Dr. Viol, it is currently believed that a logarithmic relation exists between the brightness of a radium luminous material and the time, at least in the early portion of the material's life following the initial irregularities due to the accumulation of the radio-active derivatives of the contained radium. Observations made at the Bureau of Standards do not accord with this belief, but indicate that the relation when the material is continuously protected from light is a hyperbolic one, at least over a range of about 300 days. They also indicate that the various brightness-time curves for materials composed of the same zinc sulphide but with different amounts of radium all fix the same hyperbola. In the relation $B = \frac{l}{a + bt}$ where B = brightness and t = time, the value of the constant a for a given sulphide is approximately inversely proportional to the radium content per gram of material, and b is independent of this content. The constant b, characteristic of the sulphide, determines the luminous life of the material when protected from light. The value of a per unit of radium content (a_1) is also a characteristic of the sulphide, and measures its sensitivity to *alpha*-ray excitation. At present we are unprepared to state what relation, if any, exists between the values of these constants.

DR. C. H. VIOL (*Communicated*): Dr. Kunz's patents on luminous compositions are well known to Mr. Kammer and myself—so well, in fact, that I would point out that Dr. Kunz is in error in saying they were granted in 1903. As a matter of fact, while the applications were filed in September and December, 1903, the patents were not granted until May 16, 1905, after rejections and many amendments to the original applications. So far as the

[3] Director of Research, Chile Exploration Co., New York City.
[3] Chief Chemist, Welsbach Light Co., Gloucester, N. J
[4] Bureau of Standards, Washington, D. C.

writers are aware, Dr. Kunz has made no great efforts to follow up the development and improvement of radium luminous compounds, and his patents on luminous compositions have only served to impede in the United States an industry that has advanced far in European countries.

Dr. Kunz originally sought a patent on (Claim 1, Application 186,381, issued as Patent 789,812) "A luminous composition containing a radio-active material and a radio-active responsive material, substantially as described." This and other broad claims, which presumably would have given Dr. Kunz a monopoly on any discoverable self-luminous mixture consisting of any radio-active substance and any radio-responsive materials, was rejected by the examiner, after the patent had been allowed, because the examiner discovered that in a lecture delivered by William J. Hammer before a joint meeting of the American Institute of Electrical Engineers and the American Electrochemical Society in New York on April 17, 1903,[5] the lecturer exhibited to the audience a test-tube containing a mixture of zinc sulphide mixed with radium. Dr. Kunz finally secured a patent containing several broad claims, one claim particularly, No. 11, for "A composition containing a luminescent compound, a radio-active excitant and a vehicle, of radio-active phosphorescent compounds." In view of the earlier English patents granted to Balmain and to others on the applications of phosphorescent calcium sulphide to watch dials, etc., it is difficult to see the patentable novelty of applying radio-active phosphorescent zinc sulphide to watches, etc.

It is evident then, we think, that we have done Dr. Kunz no injustice in omitting to mention his work, more particularly as we have not sought to give the impression that the development of radio-active luminous compositions was new and original. What we do claim is that we have prepared a very superior quality of phosphorescent zinc sulphide, which serves, with radium, for the preparation of a luminous composition of high luminescence.

[5] Trans. Am. Electrochem. Soc., 3, Appendix page 15.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, held in Pittsburgh, October 4, 1917, President Fink in the Chair.

SILVER PEROXIDE AND THE VALENCE OF SILVER

By H. C. P. Weber.*

[Abstract.]

The author discusses the black compound formed on the anode during the electrolysis of silver nitrate solution, and to which the formula $2Ag_3O_4.AgNO_3$ has been ascribed. He discusses the probable valence of silver in the oxide part of this formula, writing it either Ag_2AgO_4 by analogy to red lead, or $Ag(AgO_2)_2$ by analogy to magnetic iron oxide. His various electrolytic experiments lead him to conclude that the last formula, $Ag(AgO_2)_2$, in which one-third of the silver is cathodic and diatomic and two-thirds anodic and triatomic, is in harmony with his experimental results. This would make Ag_3O_4 the silver salt of an argentic acid.

The black compound which is formed at the anode, when a concentrated silver nitrate solution is electrolyzed at rather high current density, has been known for over a hundred years. Its character is, however, still imperfectly known, although it has been prepared and examined by a number of investigators. Its particular interest lies in the fact that it appears to be a type of silver compounds in which silver has a valence greater than one. As the time seems about ripe for a revision of some of our current conceptions of valence in the solid state, the valence of silver, a typically monovalent metal of the first group, is of especial importance.

In a recent communication to this Society by Mortimer J. Brown[1] a resumé of the literature on the subject, and a very careful study of the composition of the substance, is given. The

* Research Chemist, Westinghouse Electric & Manufacturing Co., East Pittsburgh, Pa.

[1] Trans. Am. Electrochem. Soc., 30, 327 (1916).

result arrived at is that the brown crystals formed at the anode may be represented by the formula $2Ag_3O_4.AgNO_3$, both the coulometer ratio and the analysis pointing to the same value.

The various oxides of silver considered in connection with the compound which has been called silver peroxynitrate, have been Ag_2O_2, Ag_2O_3, Ag_3O_4, Ag_4O_5, as follows:

Ag_2O_2.—Wallquist,[2] Fischer,[3] Mahla,[4] Mulder,[5] (in combination with O_2), Mulder and Heringa[6] and Sulc[6a].

Ag_2O_3.—Berthelot,[7] Hampe,[8] Brauner and Kuzma,[9] Luther and Pokorny.[10]

Ag_3O_4.—Mulder,[11] Mulder and Heringa,[12] Sulc,[13] Tanatar,[14] Watson,[15] Brauner and Kuzma,[16] Barbarovsky and Kuzma,[17] Brown.[18]

Ag_4O_5.—Fischer,[19] Sulc,[20] Luther and Pokorny.[21]

Most of these investigators write the formula with a certain ratio of $AgNO_3$ included, which varies somewhat with the different authors.

The dioxide formulas are mainly from the earlier periods of the literature, and have found little support in more recent work. Mulder gives the formula $3Ag_2O_2.O_2.AgNO_3$, as does Sulc, which is based upon the step-wise reduction to Ag_2O_2 by ammonia and by water. This is essentially the same, however, as $2Ag_3O_4.AgNO_3$. Luther and Pokorny have proved the existence

[2] Jour. prakt. Chem., **31**, 179 (1842).
[3] Jour. prakt. Chem., **33**, 237 (1844).
[4] Liebig's Ann., **82**, 289 (1852).
[5] Rec. Trav. chim. Pays Bas, **16**, 57 (1897); **17**, 57 (1898); **18**, 91 (1899); **19**, 115, 165 (1900); **32**, 385 (1913); **21**, 405 (1903).
[6] *Ibid*, **15**, 1, 236 (1896).
[6a] Z. anorg. Chem., **12**, 89, 680 (1896); **24**, 305 (1900).
[7] Compt. Rend., **90**, 653 (1880).
[8] Chem. Ztg., **14**, 1777 (1890).
[9] Ber., **40**, 3362 (1907).
[10] Z. anorg. Chem., **57**, 290 (1908).
[11] *Loc. cit.*
[12] *Loc. cit.*
[13] *Loc. cit.*
[14] Z. anorg. Chem., **28**, 331 (1901).
[15] J. Chem. Soc., **89**, 578 (1906).
[16] *Loc. cit.*
[17] Z. physic. Chem., **67**, 48 (1909).
[18] Trans. Am. Electrochem. Soc., **30**, 327 (1916)
[19] *Loc. cit.*
[20] *Loc. cit.*
[21] *Loc. cit.*

of Ag_2O_2 by a study of the potentials at the anode, but have not isolated the oxide.

Brauner and Kuzma considered the composition of the substance to be $Ag_2O.Ag_2O_3$, mainly from analogy to trivalent gold and copper (?). Luther and Pokorny obtain their evidence for trivalent silver from the measurement of the potential breaks in silver nitrate solution, as the step following the dioxide. For the final compound they deduce the formula $Ag_2O.3Ag_2O_3$, their analyses being made electrochemically with the small quantities deposited on the electrodes. The point of particular importance here is the fact that they obtain definite evidence for the two oxide forms, the divalent and the trivalent.

Careful quantitative analysis of the anode product in quantity, and of the processes taking place both at the anode and the cathode, agree best with the formula $Ag_3O_4.xAgNO_3$. Thus Mulder's formula and Sulc's may be written in this form. Tanatar's analyses corroborate this formula. In addition he has prepared by electrolysis of a fluoride solution the compound $Ag_{15}F_3O_{16}$, which may be written $4Ag_3O_4.3AgF$. Watson finds a similar composition. Barbarovsky and Kuzma, in a detailed study of the ratio of silver carried to oxygen liberated in a parallel coulometer, together with the changes in the nitric acid concentration at the electrodes, arrive at the composition Ag_3O_4. At the same time their results eliminate the possibility of pernitrate formation. They believe, together with Luther, that the additional $AgNO_3$ in the product is not an essential part of the formula. Finally, Brown in a very careful comparison of the copper-silver coulometer ratio, together with determinations of the composition of the crystals, decides for the formula $2Ag_3O_4.AgNO_3$.

There are a few isolated references to a possible Ag_4O_5. Sulc in his first article decides upon $3Ag_4O_5.2AgNO_3$, Fischer proposes a similar formula, and Luther and Pokorny find indications of this oxide.

Margaret Bose[22] finds an oxidation potential very close to that of Luther for Ag_2O_3, about 1.5 V., which is practically the same for sulphate and nitrate solutions.

[22] Z. anorg. Chem., 44, 237 (1905); 44, 263 (1907).

The weight of the evidence then rests with the formula Ag_3O_4. The question of the rôle of the $AgNO_3$ accompanying the substance is not so clearly answered. Most of the results agree on one silver nitrate to six silver in the oxide, but various investigators find 1:1, 1:4, and 1:5. It seems reasonable to assume that this is adsorbed material carried down from the concentrated solutions, yet its constancy and, as Brown points out, the large amount of it (20 percent) make this explanation unsatisfactory. With lower concentration of the silver nitrate in the electrolyte the coulometer ratio drops somewhat, the silver percentage not changing. This may be due to compensation by the more rapid decomposition in the more dilute solution, which would increase the silver percentage in the precipitate.

In Ag_3O_4 we must assume silver with a valence higher than two. The work of Barbarovsky and Kuzma precludes the interpretation of the substance as a pernitrate. Neither can the conception of these salts as true peroxides be maintained. They are not produced by the action of peroxides and do not yield peroxide upon decomposition. Thus a nitric acid solution of the peroxynitrate was decolorized by dilution with water and immediately poured into a titanium sulphate solution. The test did not yield a trace of the peroxide reaction. Similar results have been reported before. The compound must therefore be considered as a true mixed oxide. There are two possibilities for Ag_3O_4, one in which we have di- and tetravalent silver, Ag_2AgO_4 being formed in analogy to red lead; the second in which we have di- and trivalent silver, $Ag(AgO_2)_2$ in analogy to ferroso-ferric oxide. In either case the higher oxide should show some acidic properties, and it was to find evidence of this that the following experiments were carried out.

EXPERIMENTAL.

The black oxide formed is quite soluble in nitric acid and fairly stable in such solutions, the color being discharged only in 24 to 36 hours if the solution is kept cold. Warming or dilution with water at once decomposes the compound, hydrogen peroxide not being formed during the change. On freezing the strong nitric acid solution, which may be almost black, a yellowish crystal-mass is obtained at liquid air temperatures. This upon warming melts

to a black liquid without any evidence of separation into different constituents. The oxide which was used had been prepared by electrolysis of a nitrate solution, and consisted in part of crystals a half inch (13 mm.) long. They had been washed with dilute nitric acid until they gave no further decided evolution of gas with fresh portions of wash-liquor, and were then dried and bottled. Before use this material had been kept for eight months. Solution in nitric acid was always preceded by evolution of oxygen which soon ceased, the bulk of the material dissolving without further decomposition. Chlorides are fatal to the stability of the peroxide. Attempts to produce a similar compound to the "peroxynitrate" by electrolysis failed completely, even in very cold concentrated solutions of silver chloride in potassium chloride.

The poisoning effect of chlorides was very strikingly shown in experiments carried out to determine, if possible, differences in the Ag^+ content of peroxide and ordinary silver solutions by precipitation. For this purpose some of the brown crystals were washed with dilute nitric acid, well cooled, until little further gas evolution took place. The crystals were then dissolved in a chilled solution containing 35 percent HNO_3 and 20 percent KNO_3. This solution, which contained 0.1 g. peroxide in 20 c.c., was divided into two portions, one of which was decolorized by warming gently. Both solutions were then suspended over liquid air until they were congealed. Chlorine was added at the melting point of this mixture, in the form of the same strength nitric-nitrate solution, which contained in addition 2 mg. Cl per cubic centimeter. Both solutions gave a precipitate upon the addition of the first 0.02 mg. of Cl, no difference in the rate of precipitation upon further additions being noticeable. Not only this, but the dark peroxide solution immediately commenced to bleach and was colorless before 0.2 mg. of chlorine had been added, a quantity which was only a fraction of that necessary to convert all of the silver into chloride. This catalysis of the decomposition by chlorine reminds one somewhat of the behavior of permanganates.

No indications of double or complex salts were obtained. Ammonium salts, including the persulphate, decompose the peroxide, with the exception of the nitrate,[23] in dilute solution.

[23] This statement may require revision upon working with absolutely chlorine-free reagents. Erratic results were often obtained and were probably due to small variations in the concentration, temperature, or to impurities such as chlorine.

In concentrated nitric acid solutions ammonium salts were without effect, or very slow in discharging. Permanganate and persulphate did not interact. The only hydrogen peroxide available at the time contained chlorine, and this discharged the color at once. Chromates were without influence.

Among the heavy metals copper, mercury, lead and manganese were examined, mercury in the hope of obtaining complex or double salts. Neither one of the first two metals showed any decided reactions. Solutions of the peroxide containing mercuric nitrate appeared to have increased stability. The reactions with lead and manganese have, however, an important bearing on the constitution of the peroxide.

When a strong nitric acid solution of the peroxide is added to a saturated nitric acid solution of lead nitrate, the solution being kept well below zero, there is at first no change noticeable. In the course of 15 to 20 minutes lead peroxide begins to form, and its formation continues until the silver peroxide is used up.

With manganese salts permanganate is formed. When manganese sulphate is added drop by drop to the silver solution it is changed instantly into permanganate. The permanganate and silver peroxide are stable in the presence of each other. This oxidation proceeds with far greater velocity, for instance, than the persulphate oxidation of manganese. In parallel tests made with silver peroxide and ammonium persulphate, in which the acid concentrations were the same, the manganese was at once, in the cold, converted into permanganate, while a similar quantity of manganese showed no evidence of permanganate formation with the persulphate.

All of these facts taken together make it seem that we have in $2Ag_3O_4.AgNO_3$ silver in a high valence form corresponding to permanganates, chromates and persulphates. Chronologically the tests described, for the most part, followed the transference tests described below and were the outcome of the fact that silver may act as anion.

Electrolysis of Silver Nitrate: In the investigations described in the literature, the preparation of the peroxide has been carried on in solutions which were very nearly neutral. If a solution is used, instead, which contains 15 to 25 percent of nitric acid, the silver peroxide is not precipitated at the anode but the solution

becomes dark brown and almost black, finally. Continued electrolysis under these conditions finally produces copious oxygen evolution at the anode, but no definite substance separated, except in one case in which a small quantity of a black powder separated which it was not possible to collect.

Transference of Silver to Anode: Trial in a capillary-tube cell conclusively showed that in this dark-brown solution silver is present as an anion. A cell was prepared, as is shown in Fig. 1, consisting of a tube 180 mm. long, with upturned ends of about 20 mm. The bore of the tubing was 2 mm. At the middle an upright tube joined on permitted the solution to be introduced for examination. In this case a 20 percent solution of nitric acid was first filled in and this was followed by a solution of the peroxide in nitric acid, so that a sharp line of separation between

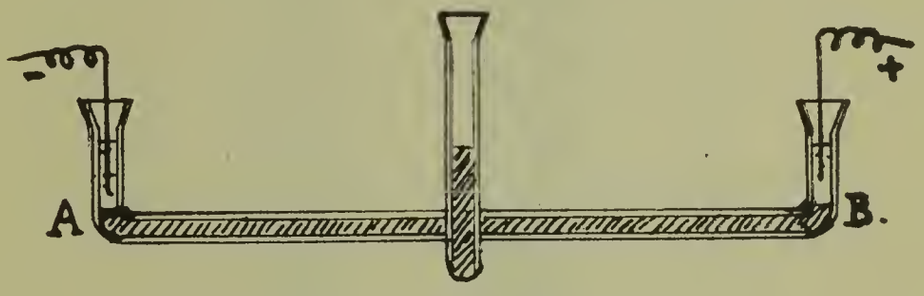

FIG. 1.

the nitric acid was formed at *A* and *B*. Platinum electrodes were then introduced at the two ends and 0.2 ampere, at 110 V., was passed through the cell. Under these conditions the dark boundary moved so rapidly toward the anode that it could be followed with the eye. At the same time a corresponding shift, due to reduction, took place at the cathode side. In ten minutes the boundary, still perfectly sharp, had moved 0.5 cm.

In order to show quantitatively that in the transference silver was actually involved, the apparatus shown in Fig. 2 was employed. The middle portion *B* is separated from the anode compartment *A* and the cathode compartment *C* by the stoppers ground into the constrictions *a* and *c*. These stoppers are hollow and terminate in the narrow tubes with funnel extending above the top of the cell. The anode and cathode respectively are supported by these tubes.

The manipulation is as follows: Some of the finely powdered silver peroxide (0.67 g.) was dropped into the middle compartment and the stoppers were inserted. A solution consisting of concentrated nitric acid, to which an equal volume of 40 percent potassium nitrate solution had been added, was thoroughly chilled and poured into the middle section, filling this and the tubes leading to it to the level *e-f*. The silver peroxide dissolved with evolution of some oxygen, to form the characteristic black solution.

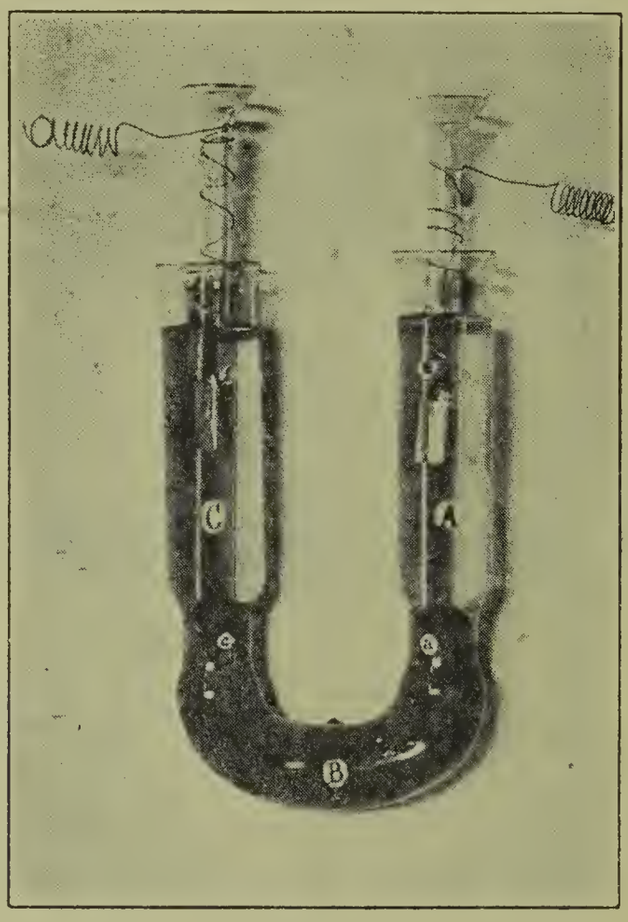

FIG. 2.

Anode and cathode compartment were then filled with the same nitric acid-potassium nitrate mixture until the level coincided with that in the inner tubes. The stoppers could then be raised and lowered without disturbing the levels of the various liquids in the system.[24] Each electrode consisted of a square centimeter of platinum foil suspended by means of platinum wire coiled around the central tubes.

[24] For the present purposes this arrangement met the requirements. For more precise manipulation the stoppers should be provided with perforations corresponding to grooves in the constrictions so that communication between the electrode and middle chambers could be established by turning instead of raising the stoppers.

The cell after filling was placed in a mixture of ice and alcohol. Current was supplied by a battery circuit at 40 V., with lamps for resistance, and was measured by a Westinghouse milliammeter. With the stoppers closed the resistance of the cell was 570 ohms, which could be decreased to 150 ohms by freeing the stoppers completely. This permitted of very convenient adjustment of the current strength by changing the clearance between stopper and grinding. The current was set at 0.15 ampere in the evening, the apparatus placed in an ice chest and left for 17 hours. The movement of the dark zone toward the anode soon became apparent under these conditions. At the end of the run in the morning the anode compartment was completely filled with the dark solution, "peroxynitrate" had not been deposited on the anode but oxygen was being liberated.

The stoppers were closed to isolate the different parts of the cell, and anode and cathode liquors were removed separately with a pipette, nitric acid-nitrate mixture being used as wash solution. The two compartments were refilled with more of the same solution and electrolysis continued.

The solutions to be analyzed were run into standard sodium oxalate solution, which was titrated back with permanganate. The silver content was then determined with standard hydrochloric acid. The results are only intended to show the character of the transfer, since in the first run there was some evolution of gas; further, the data do not include the part played by the other ions of the solution in the conduction. They do show beyond doubt, however, that silver was transferred to the anode together with the available oxygen.

With the first 9,180 coulombs passing through the cell, 0.062 g. of silver together with a minimum of 0.033 g. available oxygen were carried to the anode. At the same time 0.024 g. of silver went to the cathode.

In the second part of the determination 1,080 coulombs, at the rate of 0.20 ampere, carried 0.008 g. of silver and 0.003 g. available oxygen to the anode; 0.003 g. silver went to the cathode.

In the third experiment it was noted that although the central portion of the cell was still brown, there was little evidence of transference. This was confirmed by the analysis. For 2,250

coulombs which had passed, at the rate of 0.25 ampere, there was no silver at the anode nor available oxygen, and 0.015 g. silver at the cathode.

At the end of the third experiment the middle chamber was found to contain only 0.009 g. of available oxygen with the remaining silver. With the exhaustion of the peroxide, therefore, the transference to the anode ceases. The complete data are recorded in Table I.

TABLE I.

Expt.	Time (hrs.)	Coulombs.	Anode Ag.	Anode O.	Cathode Ag.
			g.	g.	g.
1	17.	9180.	0.062	0.033	0.024
2	1.5	1080.	0.008	0.003	0.003
3	2.5	2250.	0.0	0.0	0.015
Total....	21.0	12510.	0.070	0.036	0.042

The figures show clearly the transfer of silver as an anion, in harmony with the reactions of the substance and the effects visible under the influence of an impressed voltage, and that the effect runs parallel with the available oxygen content of the solution. In the first two experiments the ratio between silver traveling to the cathode and to the anode is 1:2.6, which is roughly in accord with a compound in which two-thirds of the silver is anodic. This supports the conception that the so-called silver peroxynitrate is the silver salt of an argentic acid in which silver has a valence of three (or four). A possible formula in harmony with all of the information available is $\overset{++}{Ag}(\overset{+++}{AgO_2})_2$ in which silver has a valence of two and three. The arrangement $\overset{++}{Ag_2}\overset{++++}{AgO_4}$ does not seem to fit the facts so well.

More detailed experiments along this line, to determine if possible whether the negative ion contains di- or trivalent silver, and whether the NO_3 always found with the solid compound is an essential portion of the negative ion, seem desirable.

SUMMARY.

1. Experiments directed towards the preparation of salts in which silver has a valence of more than one are described. With the exception of the peroxynitrate already known, the results are negative.

2. Conditions affecting the stability of this compound, such as temperature, presence of concentrated acid or salt solutions, effect of chlorine, are set forth.

3. It is shown that in concentrated nitric acid the compound is formed in solution and not on the electrode.

4. Characteristic reactions of the peroxide with lead and manganese are described.

5. It is shown that at least a part of the silver in this compound is carried to the anode during electrolysis.

6. The conclusion is drawn that the black compound previously described in the literature is the silver salt of an unstable argentic acid.

7. A convenient apparatus for the determination of transference numbers is described.

Westinghouse Research Laboratory,
East Pittsburgh, Pa.

DISCUSSION.

PROF. W. D. BANCROFT[1]: I am much interested in anything that helps straighten out this problem. It seems to me Mr. Weber has done an excellent piece of work.

There is one possible source of error which I imagine Dr. Weber took into account, but did not mention in his paper. If his black solution was colloidal, that is, contained the peroxy-

[1] Professor of Chemistry, Cornell University.

nitrate in suspension, then his experiments would mean nothing in regard to the transference of silver. His conclusions are perfectly sound if he was dealing with a true solution.

H. C. P. WEBER: I cannot give definite assurance on that point. That is, no direct experiments with this in view have been carried out. Prof. Bancroft has personally called my attention to this point, and the only way to decide it would be by ultra-microscopic or similar examination of the solution. There are, however, a number of points indicating the correctness of my position. In the first place, in colloidal solution we may have transference either to the anode or cathode, so that the probability of this anode being colloidal is one-half. The speed of reaction of this material upon reduction, which is instantaneous, does not give any evidence of colloidal nature. This, of course, is corroborative, although not binding, evidence. The solution is formed from a crystalline material, a black crystalline peroxynitrate, by the action of a strong nitric acid, and these conditions are not favorable to the formation of colloidal solutions.

On the other hand, these peroxynitrate solutions have been frozen in liquid air without evidence of any segregation. The frozen material is a yellow, almost white, crystalline mass, and upon liquification there is a gradual change in color from light to dark as the material becomes warmer; this, again, without any evidence of segregation. There are a number of other points against regarding this material as colloidal; in fact, my whole experience with the compound is against such a supposition. The definite answer would only be found if the ultra-microscopic examination showed colloidal particles.

G. C. GIVEN[2]: I think it quite likely that this material is a colloid. That was the first thing which came to my mind. If it were, I do not see that an ultra-microscopic examination would prove it were not, because a great many colloid particles are not visible in the ultra-microscope. Some as large as 30 milli-microns are not visible. I have also seen a great many colloid solutions which have been frozen, and when thawed out again were as

[2] Chemist, Atlas Powder Co., Tamaqua, Pa.

good colloid solutions as they were before freezing. I think this material could be tested by ultra-filtration.

A very simple ultra-filter can be made by pouring 10 percent collodion solution into a test-tube, inverting the test-tube until no more will drain out, blowing air gently into the test-tube until the odor of ether has disappeared, but that of alcohol still remains, and then removing the film from the inside of the tube by the addition of water. A one-holed rubber stopper containing a funnel can then be inserted into this test-tube-shaped film of collodion, and a very rapid ultra-filtration carried out. I have seen colloidal iron oxide filtered as clear as water by this means. Ultra-filtration would be the proper test, since neither freezing nor the ultra-microscope would prove it, and a colloid particle will migrate either to the anode or cathode according to its charge.

MR. WEBER: For my purpose, as far as the major argument is concerned, it is immaterial whether this is a colloid or not. The proposal to treat this solution by ultra-filtration with a collodion membrane is beyond hope of realization because a water solution is quite unstable and even rather dilute nitric acid solutions rapidly decompose while standing. In a finely-divided porous septum the material would immediately decompose. I would like to ask the question what this material would be even if it were admitted to be a colloid?

MR. GIVEN: I do not know. I do not believe that it would be silver.

MR. WEBER: That is sufficient for the purpose as far as the higher valence of silver is concerned. When you electrolyze a slightly acid silver nitrate solution and thus form a peroxynitrate you obtain it as an anode deposit which, when dissolved in strong nitric acid, gives the brown-colored solution. If, however, you electrolyze in the strongly nitric acid solution you obtain directly the deep brown solution from which, upon occasion, I have observed fine black crystals depositing, which, however, I was not able to collect. This, again, is not absolutely binding evidence against the supposition that the material is a colloid, but the for-

mation of a stable colloidal solution in the presence of considerable mineral salts and of strong nitric acid is, to say the least, unusual. The solution contained 20-30 percent of potassium nitrate. I freely admit that the question is open, so far as the evidence of this paper is concerned, but I do not have any great doubts that the transference is actually that of a silver anion.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

EFFECT OF LITHIUM UPON THE CAPACITY OF THE EDISON STORAGE BATTERY

By L. C. Turnock.*

[Abstract.]

Comparative tests of capacity of Edison storage batteries with 0, 10, 20, 30, 40 and 50 grams per liter of lithium hydrate added to the usual 21 percent caustic potash electrolyte. Test runs showed increased capacity up to 12 percent with the largest amount of lithium hydrate, in spite of the fact that the addition increased the electrical resistance of the electrolyte 21 percent.

During the experimental and commercial development work upon the Edison storage battery it was discovered that additions of lithium hydrate to the potassium hydrate electrolyte caused an appreciable increase in the available capacity of the battery. The following investigation was undertaken to determine the magnitude of the effect of various additions of lithium hydrate to the electrolyte.

For the purpose of reducing the amount of electrolyte required for the different test runs a special miniature test cell was constructed from the small pockets and tubes containing the active materials of the negative and positive plates, respectively, of the ordinary commercial size cell. These were properly insulated from one another by hard rubber and inserted in a nickel container, which was air-tight with the exception of a very small hole in the top serving for the escape of the gases generated in the cell during charge and discharge.

Prior to making the recorded runs the cell containing electrolyte

* Carnegie Institute of Technology, Pittsburgh.

with no lithium hydrate was subjected to a series of about twenty charges and discharges for the purpose of bringing it up to its maximum capacity. A charge and discharge rate of seven hundred and fifty milliamperes was employed; corresponding to a current density of approximately two and one-half to three times that recommended as normal for the commercial size units. All charge periods were of four hours duration, which was ample for a complete charge. All capacities were measured to the time the terminal cell potential reached nine-tenths of a volt when discharging at a rate of seven hundred and fifty milliamperes. During the forming test runs the cell was frequently discharged to a terminal voltage of five-tenths. Experience has shown that a new cell will come up to capacity in less time if the discharge is carried down to five-tenths of a volt or even to zero potential.

During all runs the cell was maintained at a temperature of 30° C. A constant temperature is necessary in order to secure comparable results. A lowering in temperature would increase the internal resistance of the cell, with the result that the cell would reach at an earlier period the predetermined cut-off voltage of nine-tenths, to which the capacity is measured. Obviously, this would indicate a capacity lower than the correct value. Conversely, a rise in temperature would have the exactly opposite effect.

Before the sharp advance in the price of potash a few years ago the manufacturers employed as electrolyte for their cells a twenty-one percent solution of potassium hydroxide containing fifty grams of lithium hydrate per liter of finished electrolyte. Such a concentration represents practically a saturated solution of lithium hydrate in the specified strength of potassium hydrate at a temperature around 25° C. For the recorded six test runs electrolytes were made up as follows:

Test 1—4.49 N.KOH (equivalent to a 21 percent solution)
Test 2—4.49 N.KOH in which is contained 10 g. LiOH per liter
Test 3—4.49 N.KOH in which is contained 20 g. LiOH per liter
Test 4—4.49 N.KOH in which is contained 30 g. LiOH per liter
Test 5—4.49 N.KOH in which is contained 40 g. LiOH per liter
Test 6—4.49 N.KOH in which is contained 50 g. LiOH per liter

Tables I and II show data taken on test runs one to six inclusive. Table III shows the condensed and computed results from tests one to six inclusive. Attention is directed to the last column of

Table III showing how the capacity increases with increased content of lithium hydrate in the electrolyte. A twenty-one percent solution of potassium hydroxide containing fifty grams of lithium hydrate per liter as electrolyte shows an increase in capacity of twelve percent over a twenty-one percent solution of potassium hydroxide containing no lithium hydrate. Fig. 1 illustrates the effect of lithium hydrate upon the capacity of the cell and is plotted from the results shown in the last column of Table III.

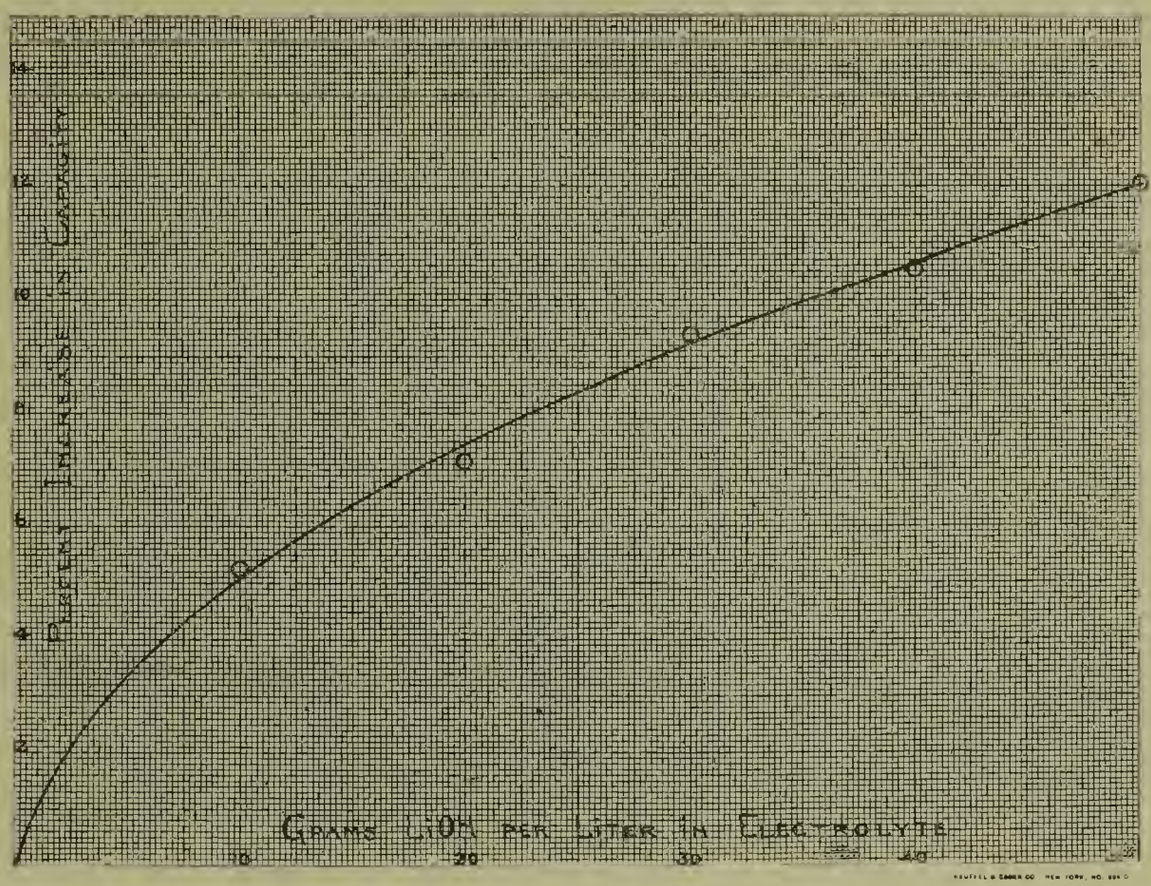

Fig. 1.

The effect of additions of lithium hydrate to a twenty-one percent solution of potassium hydroxide upon the electrical conductivity of the solution was investigated to determine whether the increased capacities shown could be accounted for through an increase in conductivity. Reference to Fig. 2 shows that additions of lithium hydrate to the potassium hydrate solution decrease the conductivity of the latter, a fact which is entirely in accord with what may be expected from the standpoint of the mass law. That additions of lithium hydrate to twenty-one percent potassium hydroxide solution increase the electrical resistance of the latter is also indicated in Tables I and II. Reference to the potential

columns of each run show how the terminal potential on charge rises at the end of charge from a value of 1.835 in test 1 for an electrolyte containing no lithium hydrate gradually up to a value of 2.03 in test 6 where the electrolyte contained fifty grams of lithium hydrate per liter. Inasmuch as a decrease in available capacity necessarily results from an increase in the internal resistance of a cell, it is interesting to observe that it is possible to increase the capacity of the cell by additions of lithium hydrate to a value of twelve percent in spite of the fact that such additions simultaneously increase the electrolyte resistance of the cell by twenty-one percent.

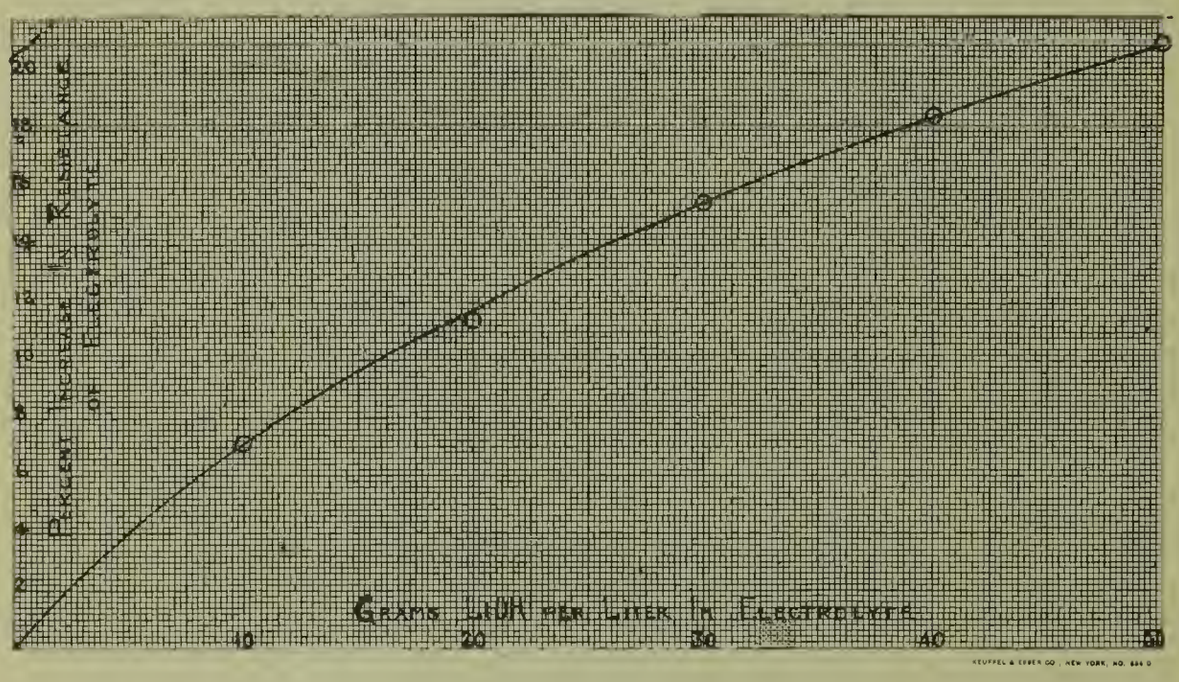

Fig. 2.

The exact phenomenon of the effect of lithium hydrate in increasing the capacity of the cell is not definitely known as far as the author is aware. More lithium appears to enter the plates on discharge than comes out on charge. In order to meet such a condition more lithium hydrate is added to commercial cells before placing them on the market than is required to maintain a saturated solution of lithium hydrate in the potassium hydrate, the excess depending upon the size of the cell and the amount of electrolyte it will require. The small cell known as the B-2 type contains about 0.45 liter of electrolyte and 9.2 grams of solid lithium hydrate crystals right after assembly, while the commercial size vehicle cell A-4 contains 1.40 liters of electrolyte and 37 grams

TABLE I.

Charging and Discharging Rate.....750 Milliamperes
Temperature ..... 30° C.

TEST 1.		TEST 2.		TEST 3.	
Electrolyte—21% KOH		Electrolyte—21% KOH containing 10 grams of LiOH per liter		Electrolyte—21% KOH containing 20 grams of LiOH per liter	
CHARGE		CHARGE		CHARGE	
Time	Terminal Voltage	Time	Terminal Voltage	Time	Terminal Voltage
9.00	1.60	9.05	1.62	8.40	1.60
9.20	1.71	9.20	1.72	9.00	1.72
9.40	1.72	9.40	1.721	9.20	1.74
10.00	1.73	10.00	1.723	9.40	1.75
10.20	1.73	10.20	1.74	10.00	1.76
10.40	1.74	10.40	1.745	10.20	1.77
11.00	1.78	11.00	1.79	10.40	1.793
11.20	1.795	11.20	1.842	11.00	1.83
11.40	1.835	11.40	1.85	11.20	1.86
12.00	1.835	12.00	1.85	11.40	1.86
12.20	1.835	12.20	1.85	12.00	1.86
12.40	1.835	12.40	1.85	12.20	1.852
1.00	1.835	1.05	1.848	12.40	1.85
DISCHARGE		DISCHARGE		DISCHARGE	
1.00	1.40	1.05	1.40	12.40	1.40
1.03	1.30	1.25	1.24	12.45	1.285
1.10	1.26	1.45	1.20	12.50	1.26
1.20	1.24	2.05	1.168	1.00	1.23
1.40	1.205	2.25	1.120	1.20	1.18
2.00	1.18	2.45	1.075	1.45	1.138
2.20	1.14	3.05	1.00	2.00	1.10
2.40	1.10	3.10	0.98	2.20	1.06
3.00	1.015	3.15	0.96	2.40	0.991
3.13	0.90	3.20	0.93	2.45	0.971
		3.25	0.90	2.50	0.952
				2.55	0.935
				3.00	0.910
				3.02½	0.90

TABLE II.

Charging and Discharging Rate.....750 Milliamperes
Temperature ..... 30° C.

TEST 4.		TEST 5.		TEST 6.	
Electrolyte—21% KOH containing 30 grams of LiOH per liter		Electrolyte—21% KOH containing 40 grams of LiOH per liter		Electrolyte—21% KOH containing 50 grams of LiOH per liter	
CHARGE		CHARGE		CHARGE	
Time	Terminal Voltage	Time	Terminal Voltage	Time	Terminal Voltage
8.40	1.59	8.20	1.59	8.30	1.66
9.00	1.74	8.40	1.76	8.50	1.775
9.20	1.75	9.00	1.75	9.10	1.775
9.40	1.76	9.20	1.765	9.30	1.785
10.00	1.77	9.40	1.778	9.50	1.82
10.20	1.795	10.00	1.78	10.10	1.875
10.40	1.82	10.20	1.79	10.30	1.93
11.00	1.875	10.40	1.875	10.50	2.00
11.20	1.878	11.00	1.88	11.10	2.048
11.40	1.873	11.20	1.89	11.30	2.06
12.00	1.87	11.40	1.89	11.50	2.055
12.20	1.86	12.00	1.89	12.10	2.04
12.40	1.858	12.20	1.88	12.30	2.03
DISCHARGE		DISCHARGE		DISCHARGE	
12.40	1.42	12.20	1.44	12.30	1.42
12.45	1.30	12.25	1.315	12.35	1.205
12.50	1.278	12.30	1.286	12.40	1.19
1.00	1.243	12.40	1.246	12.50	1.17
1.10	1.212	1.00	1.182	1.10	1.14
1.20	1.185	1.20	1.130	1.30	1.10
1.40	1.14	1.40	1.08	1.50	1.06
2.00	1.095	2.00	1.03	2.10	1.018
2.20	1.047	2.20	0.975	2.30	0.968
2.40	0.986	2.25	0.96	2.35	0.959
2.45	0.972	2.30	0.95	2.40	0.942
2.50	0.96	2.35	0.935	2.45	0.930
2.55	0.94	2.40	0.921	2.50	0.921
3.00	0.922	2.45	0.907	2.55	0.908
3.05	0.901	2.47	0.900	2.59	0.900
3.06	0.90				

TABLE III.

Test Run	Electrolyte		Temp. deg. C.	Charge Rate in Milliamperes	Discharge Rate in Milliamperes	Length of Charge in minutes	Length of Discharge in minutes	Input on Charge—milliampere hours	Output on Discharge—milliampere hours	Current Efficiency percent	Percent Increase in Capacity. Test Run equal unity
	KOH percent	Gram LiOH per liter									
1	21	0	30	750	750	240	133	3,000	1,662.5	54	Unity
2	21	10	30	750	750	240	140	3,000	1,750	58.3	5.27
3	21	20	30	750	750	240	142.5	3,000	1,781	59.3	7.14
4	21	30	30	750	750	240	145.5	3,000	1,819	60.6	9.39
5	21	40	30	750	750	240	147	3,000	1,838	61.25	10.53
6	21	50	30	750	750	240	149	3,000	1,862	62	12.03

of solid lithium hydrate crystals after assembly. Enough solid lithia crystals are put in to maintain the capacity of the cell for about a year or so in regular service. If the capacity falls off at the end of such a period it is recommended to secure additional electrolyte from the manufacturer. A falling off in capacity after a long period of use may also be due to a partial carbonation of the electrolyte. This is very apt to be due to negligence in keeping the small valve and valve seat on the top of the container clean of incrustations from the electrolyte, which is carried up mechanically in the gas spray. The water evaporates slowly leaving the solid hydrates of potassium and lithium partially exposed to the atmosphere, resulting in the formation of the less soluble carbonates, preventing the proper seating of the valve, and finally in a more rapid carbonation of the electrolyte and subsequent lowering of the available capacity of the battery.

SUMMARY.

Additions of lithium hydrate to a twenty-one percent aqueous solution of potassium hydroxide such as is employed as electrolyte in the alkaline storage battery, increases both the available capacity of the battery and its internal resistance.

The increase in capacity appears to be a direct function of the amount of lithium hydrate present in the electrolyte.

A cell employing as electrolyte a twenty-one percent solution of potassium hydroxide containing fifty grams of lithium hydrate per liter (a concentration corresponding to a saturated solution at about 24° C.) shows twelve percent higher capacity and twenty-one percent higher electrolyte resistance than one employing as electrolyte a twenty-one percent solution of potassium hydroxide containing no lithium hydrate.

Carnegie Institute of Technology,
Schenley Park, Pittsburgh.

DISCUSSION.

W. D. BANCROFT[1]: I would like to ask Mr. Turnock whether he knows that the lithium hydroxide increases the capacity of

[1] Professor of Chemistry, Cornell University.

both electrodes, or whether it is chiefly at one of them. I was interested in the title of the paper, and I was hopeful that Mr. Turnock would tell us why lithium hydroxide acts as it does.

L. C. TURNOCK: At present I am not prepared to advance an explanation accounting for the peculiar behavior of lithium hydrate in increasing the capacity of the cell. From my experience with the cell it appears that the positive or nickel electrode receives the benefit of increase in capacity due to the presence of lithium hydrate. No data corroborating this view are given in the present paper, owing to the fact that the opening in the top of the cell under examination was too small to permit of the insertion and use of an auxiliary electrode. Tests upon other cells, however, seem to support this view.

WM. R. CHEDSEY[2]: May I ask whether the addition of the lithium hydrate has any effect upon the energy efficiency as well as the ampere efficiency and capacity? We would expect the energy efficiency to be increased when the ampere efficiency rises, but the increase in resistance may annul or offset this. I would be interested in knowing if Mr. Turnock has any data on the energy efficiency.

I have attempted some experiments to determine the energy efficiency, without LiOH, and with 50 grams per liter of LiOH, and also to determine the "why" of the action of the lithium hydrate. These experiments have not yet given results that I can interpret and publish, but may in a few weeks.

[2] Associate Professor of Mining, State College, Pa.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 4, 1917, President Fink in the Chair.

SUGGESTED REFORMS IN SOME CHEMICAL TERMS

By Carl Hering.*

[Abstract.]

Some of the common terms used in chemisty are shown to be indefinite, inadequate, inconsistent, and not in accordance with modern developments in physical chemistry; suggestions are made how their definitions and scope could be modified and their meanings strictly defined, so as to bring about a much desired definiteness, adequacy, consistency, a more complete concordance with the teachings of physical chemistry, and a simplification of their conception by the student. The paper is in part a revised summary of a number of articles published recently by the writer.

Physical chemistry has done so much for the benefit of chemistry that chemists should in return reorganize, readjust and redefine some of their terms to bring about a better concordance with physical chemistry and a better consistency in pure chemistry; in other words, a house-cleaning in chemical terms is in order. The purpose of the present paper is to point out some of these deficiencies and to suggest a few reforms which would help to systematize these terms, make them more consistent with each other and with modern developments, and to simplify the conceptions involved. The scope of the paper is limited to that branch of chemistry in which electrons are involved; no attempt is here made to embrace also organic chemistry and the chemistry of non-electrolytes.

*Consulting Electrical Engineer, Philadelphia.

This paper is in part a revised summary of recent publications[1] of the writer.

Bonds:—When H_2O is electrolyzed into its two gases, each of the two bonds which connected them has been broken. It is clearly understood that each H atom was combined by one bond (valence 1) and each O by two (valence 2), yet the resulting gases are both diatomic, which means that two atoms are combined to form one molecule, hence there is now a new bond between each two atoms of H and a new one between each two atoms of O. Each gram-atom of H had only one bond to begin with, as its valence is 1, and that bond has been broken, yet one still exists; to say that it originally had two bonds, therefore a valence of 2, only one of which was broken, would result in worse confusion. The O had two bonds to begin with, as its valence is 2, both of which were broken, yet one still exists; to say that it therefore had a valence of 3 would lead to great confusion and meet with a storm of opposition. Each electrolytic bond corresponds to a known and definite charge, while the others do not. Moreover these new bonds between atoms of the same element can never be broken electrolytically or by ionization, as it seems impossible to set free the same element simultaneously at the two electrodes. And each electrolytic bond must be considered to be electrically positive at one end and negative at the other, hence one of the two H's in the molecule H-H would have to be negative, and as the bonds of H are always positive, still greater confusion would arise. The formula for H_2O_2 is generally written H-O-O-H in which the bond joining the two O's is of the non-electrolytic kind like that in the diatomic molecule O-O, yet is now considered to be one of those included in saying that the valence of O is 2; this still further adds to the confusion.

It is therefore very desirable, at least in electrochemistry, to avoid such confusion by making a distinction between these two kinds of bonds, as for instance by the terms electrolytic bonds and non-electrolytic bonds. The former may also be broken by other

[1] "Inadequacy and Inconsistency of Some Common Chemical Terms." Met. and Chem. Eng., Dec. 1, 1916, p. 649.

"Oxidation and Reduction in Physical Chemistry. Consistency of Terms and Conceptions." *Ibid.*, May 1, 1917, p. 507.

"Elements Neither Free nor Combined. A Third State." Jour. Amer. Chem. Soc., August, 1917, p. —.

"Standard Table of Electrochemical Equivalents," published in book form.

than electrical means, as by heat alone, but they at least may be broken electrolytically, while the others never can. They are evidently of a physically different nature, and each should therefore have a distinctive name.

An electrolytic bond is always represented as being positive with respect to one of the two elements of the combination and negative with respect to the other, yet it is the same bond; hence the common terms positive and negative bonds are not strictly correct unless it is understood that the signs refer to the respective ends of the same bond, when it is symbolized by a line.

Every electrolytic bond, no matter which elements it combines, represents electrically 96,494 coulombs (one faraday) when the chemical symbol represents a gram-atom; and when broken it will be replaced by a free, positive charge of that many coulombs on one of the elements and a free negative one of that many coulombs on the other. The strength or affinity of these bonds is measurable in volts which are however not the same in all cases; these bonds therefore have definite though different energy values. They are always included in the valence, while the non-electrolytic bonds unfortunately are sometimes included and sometimes not, which is another inconsistency. These electrolytic bonds therefore have certain clearly known physical properties by which they can be definitely distinguished from the other kind which do not have them, though both may have other properties in common.

Valence:—This important term is used in such different senses by different writers that its utility suffers. Most chemists use it as representing the number of bonds *per atom* of an element in combination, and the values in this sense are given in tables; yet others say that in Na_2SO_4 for instance, the Na has a valence of 2, and the O a valence of 8, yet in tables giving the ordinary valences of the elements those values are never given for those elements; those who use the term valence in this sense mean the number of bonds existing in that combination, and not those per atom, hence either they are wrong or else the usual tables are wrong; both cannot be right if the term valence has any definite meaning.

In view of the fact that such tables of valences are in common use, it is here recommended to limit this term valence to the number of bonds *per atom,* the sense in which it is used in such tables;

and if one wishes to refer to the number of bonds in a compound then why not say so by calling them what they are, namely bonds, and not confuse matters by using the term valence in two different senses.

Some writers use the older term valency as synonymous with valence. It is recommended here to make good use of a distinction between them by letting the term valency be used to represent the *property* of an atom of that element to combine with others in those proportions, and limit the term valence to the *one particular value* which it has in the specific case; thus it would be said that copper as an element possesses the property of having a *valency* of 1 and 2, and that in cuprous salts it has a *valence* of 1 and in cupric salts a *valence* of 2.

To thus limit the term valence to the actual number of bonds per atom in a particular compound, also has other important advantages. In that sense when an element is in its free state it of course has no bonds connecting it with any other element, hence it must follow as a necessary consequence and as a matter of consistency, that its valence in its free state must be zero; the utility of this will be shown below. Some writers use the term zero valence as referring to those elements (like the noble gases, such as argon) which never enter into any combination. It is recommended here to use the term non-valent in such cases, and let the term zero valence always mean the uncombined state of any element. All chemical reductions are always reductions of the valence, and when the last one of the bonds has been broken there naturally can be none left, and the element is then reduced to its free state, hence a free element must necessarily have a zero valence. Moreover the valence of some elements may change from + to — or the reverse; it must therefore follow as a necessary mathematical consequence that they then must pass through a zero valence.

Most elements will during electrolysis always go to one particular electrode and never to the other; hydrogen for instance always goes to the cathode and oxygen always to the anode. Some others, however, like chlorine, nitrogen, sulphur, etc., sometimes go to the cathode and sometimes to the anode. The direction in which they go is determined by the sign of the electric charges carried by them, and as every 96,494 coulombs (one faraday) of such

charges corresponds to one of the bonds of the valence (of a gram-atom), it will often simplify and clarify matters to attach these signs to the valences, that is, to distinguish between positive and negative valences; although recommended long ago[2], this is not usually done; it would in many cases simplify and clarify conceptions and calculations, besides bringing about better consistency.

Attaching these signs to the valences not only shows which electrode the respective elements will go to, that is, whether they carry + or — charges, but also is of use in bringing about consistency in some cases in which inconsistency and confusion now exist, it is in fact inconsistent not to do so. For instance in oxidizing the sulphur in H_2S to SO_3 the increase of valence is really 8 units because the valence of S in H_2S is really —2 and that in SO_3 is +6, hence the total change (the algebraic difference) is 8 and not 4 as it would appear to be when the negative sign is neglected. The sign of this change of valence indicates the direction of the current which would bring about this change, if electrolytic; if the sign of this change is negative it involves a reduction, if positive, an oxidation. Changing CH_4 (methane) into CCl_4 (carbon tetrachloride) is acknowledged to be an oxidation; but it is also universally acknowledged that oxidation is always an increase of valence and yet the valence of C in both is 4, that is there has been no increase. If however the proper signs are attached to these valences that of C in CH_4 is —4 while in CCl_4 it is +4, it is at once clearly evident that there has been an increase of 8 units in the valence, the algebraic difference; and if this chemical change could be produced electrically it would be found to require 8 faradays or unit changes or enlarged electrons, to do it, for each gram-molecule.

When the signs of the valences are included the sum of the positive and negative ends of the bonds (or the charges, after ionization) must necessarily always add up to zero; thus in H_2SO_4, $+2 +6 -8 = 0$. This enables the sign of any one of them to be determined when those of the others are known.

Every electrolytic reaction involves a change of valence, there is always an increase of valence in the anions, as is manifested at

[2] See the writer's article in Electrochem. Industry, now Met. and Chem. Eng'ng, January, 1903, p. 169.

the anode, and a decrease in the cations, as is manifested at the cathode; and each unit change of valence corresponds to a charge of one faraday (96,494 coulombs or 26.80 ampere-hours). To neglect the negative signs of the valences therefore introduces such confusion and inconsistencies that this very useful rule and guide becomes useless, while when the negative signs are attached to the valences to which they belong, this rule becomes one of the most useful and helpful in electrochemistry, as it is rigidly correct, it being a modernized form of expressing Faraday's law.

Change of Valence:—It should be more clearly brought out that the valence of an element in combination is not the basis of calculations of the quantities of material involved in electrolysis and in the ionization which precedes it. It is the change of valence and not the valence itself which is the basic factor. Whenever an element is set free from a compound the change of valence and the valence which it had, are the same, as the valence of a free element is zero. But it not infrequently happens that nothing is set free, as for instance in the depolarizers of batteries, in the electrodes of storage batteries, in the reduction of ferric to ferrous compounds, etc.; in all such cases it is the change of valence and not the valence itself which is the basic factor in calculations. Iron in ferric compounds has a valence of 3 and in ferrous 2, hence the change of valence is 1, neither of the valences 3 nor 2 would give the correct result.

Electrons:—As it is the fashion today to talk in terms of electrons which constitute these electric charges, and as these electrons are so inconceivably small and of such indefinitely known size that they cannot yet be used in practical calculations, the writer recommends[3] introducing a very definite, practical unit for measuring quantities of electrons which might be called the enlarged or practical electron or a gram-atom-electron, and which is exactly equal to the faraday (96,494 coulombs or 26.80 ampere-hours). It then becomes strictly correct to say that each of the bonds represented by the valences (as valences are defined above) corresponds to one of these enlarged electrons, and that therefore the gain or loss of 1, 2, 3, etc., units of valences involved in oxidations and reductions,

[3] "An Enlarged Electron of Practical Size: The Faraday." Met. and Chem. Eng., May 15, 1917, p. 598.

corresponds with a gain or loss of 1, 2, 3, etc., of these enlarged electrons respectively, for each gram-atom or gram-ion of the material.

Elements neither free nor combined:—Modern teachings that elements are always in either the free or the combined state, and in no other, has led to considerable disagreement and confusion as to which of these two states an element is in when in the ionized state. It is apparently not disputed that when an electrolytic salt is dissolved in water it is dissociated into two kinds of ions carrying electric charges of opposite signs, and that there are then many more molecules than the original number of molecules of the undissolved salt; the salt is then said to be ionized. Some say these ions then are free, yet it is known that they are not, nor are they still combined, as the salt has separated into more molecules than there were before, yet the solution still has more of the properties of the salt than of the free elements. To call them free is therefore as incorrect as to call them combined; the adherents of each view must state it with an apology when they teach their students, and the student naturally is left with a confused idea. Physical chemists disagree as to whether those elements in this ionized state which carry the positive charges can be said to be reduced or whether the real reduction occurs only when they are subsequently freed by the current.

Under these circumstances much confusion would be avoided by recognizing this ionized state as a third state, which is neither the combined nor the free state; there is a difference in kind and not merely in degree.[4] Discussions of this state can then be more intelligently carried on without wasting mental efforts to try to identify this state with either of the others.

It is known definitely that neither the original, undissolved salt nor its constituent elements when quite free, carry any free charges of electricity, and it seems not to be disputed that while ionized they do carry such free charges. They get these free charges in some mysterious way during the process of dissociation or ionization, and they are relieved of them again in a less mysterious way during the process of electrolysis by transmitting them to the electrodes, whereby the elements become really free.

[4] "Elements Neither Free nor Combined. A Third State." Journal American Chemical Society, August, 1917, page 1619.

Reduction:—It is probably not contested that chemical reduction always is a reduction in valence as its name implies, and this ought to be its strict and correct definition. When a ferric compound is changed into a ferrous it is universally called a reduction; the valence was reduced from 3 to 2, namely by 1 unit; if the ferrous could again be reduced in valence by another unit it would still be a reduction, yet in neither case is the iron set free; hence true reduction does not necessarily mean that the element has been set free; it is only when the valence has been reduced completely to zero by the breaking of the last bond, that the element is set free; freeing an element is therefore a mere incident in some reductions, but is not an essential or governing factor in defining reduction.

Moreover as chemical reduction is necessarily a reduction in valence, an element may have its valence reduced from 0 to a negative valence, in which case the combined element has been "reduced" from its free state, as for instance when free sulphur (valence 0) enters into combination with H to form H_2S in which the valence of S is —2. Moreover an element may be set free from its combination by oxidizing it, as will be shown below.

Setting an element free from its combination is therefore not an essential attribute of reduction; the reduction of valence is what should characterize chemical reduction.

When two equal and opposite charges bind each other, the body is electrically neutral and the bond exists. When these two charges become free the bond which had held them together no longer exists, hence *free charges mean broken bonds; either the bonds or the free charges exist, they cannot both exist simultaneously, as each one takes the place of the other.* Hence when the free charges exist, as they do after ionization, the bonds must have been broken, and as chemical reduction is a breaking of bonds, that is, a reduction of their number, it must have occurred during the ionization or dissociation. The subsequent freeing of the ionized elements by relieving them of their charges during electrolysis, is therefore not the true seat of the actual reduction, if the above analysis is correct. This is contrary to the generally accepted ideas, but the writer considers it to be correct and it certainly brings about a logical consistency which did not exist before, and it simplifies the whole subject and makes it easier for the student to understand.

The process of discharging the ions should be given a name, so that it can be referred to specifically.

In modern nomenclature one or more + and — signs are often placed over the ions of a compound indicating the number of charges carried by the ions. Over the Fe in ferric compounds there are 3 plus signs and in the ferrous compound 2. From this it is argued in some books that the iron has lost one positive charge when it was reduced from ferric to ferrous. The writer however contends that this is only apparent but not real, and that the real facts are quite the contrary, namely that it has lost a negative charge during this reduction. These charges represent those carried by ionized iron *after* the former was reduced to uncombined iron from a ferric compound and the latter from a ferrous, that is, after the three and two bonds had been replaced by three and two charges respectively; and during this transformation of bonds into charges the ferric iron lost three negative charges (because it left three positive ones on the ion), and the ferrous lost only two, when both were reduced to uncombined iron by ionization; hence the ferrous iron had one less to start with, which it must therefore have lost when it was reduced from ferric to ferrous before the ionization.

It seems true that the energy involved in the freeing of the ions of their charges is greater than that involved in ionizing them, and that the former more nearly corresponds with the energy found to be involved in the purely thermal process used to determine the so-called heat of combination, which must necessarily be equal to the sum of the other two. But are we prepared to say that in this thermal process all the energy is used merely in breaking the bond? May it not be that this thermal process is also a double or two-step process, and that the breaking of the bond is merely one of these steps? We are at least not able to say that it is not so, and in view of the lack of more definite knowledge concerning the correct analysis of these energy reactions, they should not deter us from accepting what seems to be the more logical and consistent conclusion which seems to agree with all other facts, that the true reduction, namely the breaking of the bonds, occurs during the ionization or dissociation, as the latter term also implies.

The confined space above a liquid is usually saturated with the vapor of the liquid. It is known that the vaporization involves

an expenditure of considerable energy, the so-called latent heat, yet the amount of energy which must have been consumed in saturating this space with this vapor is generally so small that it is not considered. If however this vapor were removed very fast, say by a blast of dry air, the latent heat makes itself very evident by the rapid cooling, or by requiring heat enough to be added to keep it boiling, so as to keep the space above it in a state of saturation. Why is not this analogous to the energy reactions in ionization and in the subsequent discharging of the ions by electrolysis. A comparatively small amount of energy is involved in "saturating" the solution automatically with the ions; it is small because the quantity of ions is small; but when these charged ions are being rapidly removed by electrolysis the energy required to maintain this state of "saturation" becomes very large and very evident. The process of ionization may therefore in fact require considerable energy just as in vaporization. Are we prepared to say that this is not so?

Chemical reduction being a reduction in valence, must necessarily, for the sake of consistency, be applied to all cases in which there is a reduction of valence, whether from + to or toward zero, or to a negative valence. Thus in the decomposition of water the H is reduced, as its valence is reduced from 1 to 0. But in the formation of H_2S the S must also be said to have been reduced, as its valence has been reduced from 0 to —2; the same is true of the O in the formation of water from this gas; in the oxidation of an element by free oxygen the element is properly said to be oxidized, but the oxygen must be said to have been reduced, as its valence was reduced below 0; its oxidizing power was thereby reduced, as combined oxygen has less oxidizing power than when free.

Oxidation:—Reduction and oxidation are exactly the same chemical process but are merely reversed in direction; hence all that is true of reduction must be true of oxidation in the reverse sense. Oxidation is therefore always characterized by an increase of the valence or an addition to it; it is additive in character while reduction is subtractive; in a general sense oxidation is an addition of one element to another, while reduction is a subtraction of it. Any chemical reaction which does not involve either a reduction

or an increase of valence ought properly not be called either a reduction or an oxidation.

When two elements are being combined or separated, involving changes of valence, it necessarily follows that if one is being reduced thereby the other must be thereby oxidized, or the reverse. Thus when H and O combine to form water, the H is being oxidized (valence increased from 0 to 1) and the O is being reduced (valence reduced from 0 to —2), and in their subsequent separation the H is being reduced (valence reduced from 1 to 0) and the O is being oxidized (valence increased from —2 to 0). The application of these terms to the oxygen is not usual, but it is more consistent and brings about concordance; the free oxygen is then "oxidized oxygen," its oxidizing power is greater after this separation.

Gain and loss of charges:—A combination of elements before dissociation or ionization has no free charges, nor have the resulting freed elements. Hence whatever free charges they gain during ionization they must lose again during electrolysis; the two processes must necessarily balance exactly. The cations which go to the cathode during electrolysis must necessarily carry positive charges while ionized, and the anions negative ones, or else they would not move in the directions they do. The cations must therefore have gained positive charges and the anions negative ones during dissociation, and they lose them again during electrolysis.

When the above-mentioned enlarged electron or faraday is taken as the unit charge, then each charge corresponds to one unit change of valence, or to one of the bonds of each gram-atom that have been made or broken. It must be remembered however that this applies to either the positive or the negative charges, but not to both.

All true electrolytic reactions, including the ionizations, being based on the gain or loss of charges, hence of bonds or valences, belong to the class of either reduction or oxidation, and to no other. This might be of use in some cases in distinguishing primary from secondary reactions. In such reactions as the intense and strongly exothermic one $H_2O + SO_3 = H_2SO_4$, if this can be called a chemical one, there is neither a loss or gain of any bonds or valences, but merely a rearrangement of them; it could therefore not be produced electrochemically.

Modern developments in electricity compel us to recognize that it is the negative electricity which is the kind that moves, and that it therefore moves in the opposite direction to the conventional arrow; hence consistency compels us to consider the negative charge as the unit of reference in place of the positive, in speaking of the gain or loss of charges.

If the chemical reduction is admitted to take place during the ionization, that is when the bonds are being broken or reduced and converted into free charges, as explained above, then during reduction there is a loss of charges or electrons (negative ones) and during oxidation there is a gain of such charges. The cations which are the reduced ions, carry + charges, which means that they have lost — charges; the anions which are the oxidized ions (in the sense above explained) carry the — charges which they have gained; in the un-ionized compound these two charges are combined and therefore are neutral and not free, and after the ions have discharged their charges on to the electrodes the freed elements are again electrically neutral.

Adduction:—As the term oxidation is inconsistent when applied, as it often is, to combinations with other elements than oxygen, and as it does not, like the term reduction, explain itself, it is recommended to use the term adduction in place of oxidation, because the process is of an additive nature. Reduction then implies a subtraction, hence a loss of valence and negative charges or electrons, and in the more usual sense, the loss of a companion element and of bonds, while adduction then implies the exact reverse, namely an addition or a gain of valence and negative charges and, in the more usual sense, the gain of a companion element and of bonds.

Sign of Electrode Potential; an Exothermal Charging:—To use as an illustration a zinc electrode in zinc sulphate, it is known that the zinc ions, which leave the formerly neutral zinc electrode, have acquired positive charges and therefore their former companion negative charges must be left on the electrode; hence the electrode is negative to the zinc ions in the solution; negative electrons have moved from the neutral zinc atoms to the electrode, charging the latter negatively and leaving the zinc ions charged positively. If now a current be allowed to flow, as by connecting a voltmeter to

them, negative electrons must flow from the electrode to the zinc ions in that external circuit, namely in relatively the opposite direction, though in the same direction around the circuit. Or the conventional current (positive electrons) flows from the electrode to the solution in the cell and from the solution to the electrode through the circuit outside of the cell. The apparently contradictory fact that this conventional current flows from the electrode to the solution in the cell, although the electrode is negative to the solution, is due to the fact that this is a generator or originator of the current, hence is exothermic; in all generators the current must necessarily flow from the — to the + pole in the interior in order to flow from the + to the — pole in the external circuit.

Faraday's Law:—Unless Faraday's law is stated in terms of the *change of valence* instead of the valence, it is not a universal law, and in fact is not always correct, as there are numerous cases in which it is the change of valence and not the valence itself which governs the quantitative calculations, as was explained above; to base the law on the change of valence makes it universal, as it then applies to all cases.

In electrolysis, when the equivalents between the quantities of electricity (the electrons) and of the elements set free by them are given in terms of the *volumes* of these elements in their gaseous forms, instead of in terms of their weights, there exists a simpler and more fundamental law than that of Faraday, as the volume (at 0° and 760 mm.) per electron (or per coulomb or ampere-hour) is then the same for all the elements per unit change of valence when they are in their monatomic state (one atom per molecule), namely one liter per 4,309.7 coulombs, or twice this for a liter of a diatomic gas, three times for a triatomic one, etc.; or when the change of valence is more than one, this constant must be multiplied by the change of valence. This may be deduced from a combination of the laws of Avogadro and Faraday, and assumes the products to be perfect gases. It could be termed the electrophysical equivalent, as it is independent of the atomic weights or densities of the elements.[5]

This being the more basic or more fundamental physical law, Faraday's law may be considered a corollary of it.

[5] "Electrolytic Equivalents of Gases" by the writer. Met. and Chem. Eng., April 1, 1917, p. 383.

SUMMARY.

A more precise distinction should be made between different kinds of bonds. The term valence should be more strictly defined; negative and zero valences should be included. The proposed practical unit for measuring electrons should be used. The ionized state should be considered as differing in kind from either the free or the combined state. Reduction should include all and only cases of reductions in valence, and oxidation all and only cases of an increase of valence, hence the new term adduction. The significance of the gaining and losing of electrons should be more clearly understood; the sign of electrode potentials is then more easily understood. As sometimes stated, Faraday's law is not a universal one, and it is a corollary of a more basic one.

DISCUSSION.

W. D. BANCROFT[1]: I am sorry that Dr. Hering has seen fit to publish this paper in its present form. A man writing on this subject should familiarize himself with the literature. It is quite possible that with hydrogen we have non-polar bonds; that has been suggested before. But the matter cannot be settled in the charming off-hand way Mr. Hering uses, of saying we cannot have a positive and negative hydrogen in the case of molecular hydrogen.

If you start with J. J. Thompson's electron theory, your molecule of hydrogen comes into being by one of the atoms of hydrogen taking an electron from the other, in which case one of these atoms is positive, and the other hydrogen atom is negative. It seems to me J. J. Thompson's views are at least worthy of reference in a case of this sort.

Mr. Hering postulates non-polar bonds in the case of hydrogen; on the other hand, he assumes polar bonds with methane. All of the other people who believe in non-polar bonds have non-polar bonds also in the case of methane. This should have been discussed.

Mr. Hering states specifically that he limits his discussion to

[1] Professor of Chemistry, Cornell University.

electrolytes, and to things of that sort, but there is no theoretical difference between the reduction of chlorates and of nitrobenzene.

Then Mr. Hering quotes an extraordinary statement from an unnamed chemist in regard to the valence of sodium sulphate. I am perfectly certain Mr. Hering has misunderstood, and consequently misquoted, the chemist. What the unnamed chemist meant was that you have two sodium and eight oxygen bonds.

Mr. Hering says that this method has brought about a great simplification in his point of view. Of course, I cannot dispute that because I do not know what his point of view was. But there is nothing in this paper that makes a simplification, as far as I am concerned. It seems to me it makes things more complicated, rather than simpler.

J. W. Richards[2]: I think there are some points in this paper which we could study and consider with profit. I believe the conception of zero valence for the free elements is required of us as a necessary corollary of Faraday's Law of Electrolysis. The conception seems to be a useful one. It is not so stated or explained at present in the text books, but I think it is useful to consider it, and perhaps to adopt it. The idea that when an element passes from positive valence to negative valence it passes through zero valence, or the broader idea, that when freeing it you have reduced or adduced it to zero valence, is to my mind a logical and a useful conception.

There are some details of the paper with which I cannot agree. One is that we adopt a new electron of 96,500 coulombs. Thinking over that since Dr. Hering suggested it a couple of months ago, I cannot see that it means anything else than giving another name to the faraday, and there is absolutely no need of doing so. Mr. Hering says, on page 425, "When the above-mentioned enlarged electron or faraday is taken as the unit charge, then each charge corresponds to one unit change of valence, or to one of the bonds of each gram-atom that has been made or broken." He thus gives them alternatively, the one as equivalent to the other. I see no use in speaking of any new unit, since the new one is really the old unit which we have been using.

I cannot agree with Dr. Hering that when you put a substance into solution reduction takes place in the act of solution. That

[2] Professor of Metallurgy, Lehigh University.

is, I think, a fundamental error. The reduction is accomplished only by the passage of faradays of current.

On page 422, we read: "When the free charges exist, as they do after ionization, the bonds must have been broken, and as chemical reduction is a breaking of bonds, that is a reduction of their number, it must have occurred during the ionization or dissociation." If we dissolve ferric sulphate in water, to what is it reduced by the act of solution? If you say the reduction has taken place, is it to ferrous sulphate, or to iron? I cannot see that Mr. Hering's statement brings anything but confusion into our views on that subject.

I do not like Mr. Hering's argument on page 423: "May it be that this thermal process is also a double or two-step process, and that the breaking of the bond is merely one of these steps? We are at least unable to say that it is not so, and in view of the lack of more definite knowledge concerning the analysis of these energy reactions, that should not deter us from accepting it." That is not logical reasoning. We may not be able to prove that the moon is not made out of green cheese, but we do not therefore have to accept the dictum that it is.

I do not agree with Mr. Hering's statement that Faraday's Law is only a partial statement of a more general law. It is true, as stated at the bottom of page 427, that we can associate a definite amount of any mono-valent gas, which is monatomic, with a definite number of coulombs; also a definite amount for diatomic gas, for a triatomic one, etc. But it seems that Faraday's Law is more simple than this complex statement of the connection of the volume of the gas with Faraday's Law.

There are statements in the paper with which no chemists disagree; and there are statements, as Prof. Bancroft pointed out, which should not be ascribed to chemists as a class, because they are not used by careful chemists.

I think Dr. Hering has criticized the chemists mostly from the electrical engineer's point of view, without a thorough appreciation of chemical usage.

F. C. Frary[3]: At the bottom of page 422, after discussing reduction by solution, it is stated "This is contrary to the generally accepted ideas, but the writer considers it to be correct, and it

[3] Research Director, Aluminum Company of America.

certainly brings about a logical consistency which did not exist before, and it simplifies the whole subject and makes it easier for the student to understand."

I fail to see that because it is easy to understand a thing, it makes it true.

At the top of the next page, "The process of discharging the ions should be given a name, so that it can be referred to specifically." Why not call it "electrolysis," if Dr. Hering wants a different name? That is what electrolysis is. I do not see that the name is needed. It seems disadvantageous to add more new names.

With regard to zero valence, I think our conception of valence has been muddled by the different authors of text-books. Valence is the combining power of an element, measured by the number of atoms of hydrogen which one atom of that element will combine with or replace. Certain elements, whose vapor has been shown to be monatomic, may be said to have zero valence in the gaseous state. But in all other gases we do not believe that the elements have a zero valence, and there is no advantage in saying so. Where we do not know anything about the number of atoms in the molecule, as in the solid metals, etc., I do not see why we should make the unwarranted assumption of zero valence instead of the more plausible and reasonable assumption that the atoms are combined in some way or another, and must have some sort of valence in that combination. We do not know what that valence is, but we are certain it is not zero.

J. W. RICHARDS: I wish to champion Dr. Hering's position on that point. As an example: In a ferric compound the iron atom has a valence of 3. We pass 1 faraday and this reduces its valence to 2. It reduces it to a ferrous atom. We continue to pass the current, send 2 faradays more through, and remove two more valences, and it is thoroughly logical to say that we have reduced the valence of the iron atom to zero.

Mr. Frary has already said that we have reduced the valence of the iron from 3 to 2 by the passage of one faraday. When we still further reduce it, with two faradays more, we reduce it to zero. Whether you like it or not, the statement is strictly logical.

H. C. P. WEBER[4]: The idea of zero valence is not new and no objection can be raised to its application if certain limitations are kept in view. If you consider valence as a function of the electrical charges on the atom, then you are justified in saying that "A free element has zero valence." Thus, for instance, free silver has a valence of zero. In Ag^{+-} we have zero charge, or in $H^{+}H^{-}$ the same. That is an experimental fact, of course, and while the statement that the valence is zero, since the valence electrons are not functioning, is arbitrary in a way, it fits the facts of the case if we understand it as "equal to" zero.

The conception has been used for some time in text-books. Treadwell employs it in his Qualitative Analysis (4th Am. Ed., 1916), and W. A. Noyes, in his general chemistry (1914), devotes considerable space to it. In class work—I speak of work with students at the University of Illinois—I found the zero valence method of balancing oxidation and reduction equations the most satisfactory for the majority of the students. In the reaction

$$\overset{0}{Cl_2} + 2\,\overset{++}{Fe}\,\overline{Cl_2} = 2\,\overset{+++}{Fe}\,\overline{Cl_3}$$

chlorine has zero valence to begin with. A molecule loses 2 charges, each atom of chlorine taking a negative valence of 1, while two iron atoms increase from +2 to +3.

All this is not new, and with the facts clearly in mind furnishes a very useful working conception.

CARL HERING: The fact that this subject of chemical terms and definitions has given rise to so much discussion for years, is the best kind of proof that there is dissatisfaction, and therefore room for reforms and improvement. Among electrical engineers there is no such perennial discussion about the meaning of their terms and units, as they have been clearly and definitely established long ago and are consistent throughout; any faults in them were at once rectified. The reforms described in this paper are mere suggestions; I do not think anyone can deny that there would then at least be a logical consistency throughout. If these suggestions merely act as stepping-stones to something better, their purpose will have been accomplished.

I would not have written the paper had I not been encouraged

[4] Research Laboratory, Westinghouse Elec. & Mfg. Co., East Pittsburgh, Pa.

by some chemists; Dr. Bancroft's views are therefore not held by chemists in general. His criticisms are based on matters of opinion and not of fact; they are of a broad and sweeping character and therefore cannot be answered specifically. He points out no specific errors; moreover he apparently has not read the paper with care. As far as I know, nothing that I have said contradicts J. J. Thompson's views, for which I have the highest regard. Thompson is reported to have said: "an atom plus an electron is a mono-valent anion; an atom minus an electron a mono-valent cation." As this presumably refers to negative electrons, does this not mean that oxidation is accompanied by a *gain* of negative electrons and reduction by a *loss,* which unorthodox statement in my paper, and the deductions from it, are the ones that are most bitterly attacked?

As to Prof. Richards' comments about using the faraday as the practical unit for measuring electrons, if he will read a short article of mine (Metallurgical and Chemical Engineering, May 15, 1917, page 598) he will find I then made the suggestion he now makes. The purpose of that paper was to show that it would be mathematically correct to use the faraday as a practical unit of electrons, and that it would simplify engineering calculations to do so, beside being a great convenience.

As to whether chemical reduction is accompanied by a *loss* or a *gain* of negative electrons, I understand that there exists at present no direct physical proof. Until such proof is established we are limited to indirect deductions, and as my deductions seem to me to be the more rational, and a mathematical consequence, I have at least just as much right to hold them as others have to hold the opposite view, until a definite physical proof has been established. We must, however, recognize that it is the negative electrons which travel in the opposite direction to the flow of the current indicated by the conventional arrow.

If instead of jumbling together all kinds of chemical bonds having different properties we separate them into at least two classes, because we can then formulate definite properties of at least one of these classes, I claim that this is a step toward a better system, especially as the latter class is a very important one. My opponents refuse to make this classification; this is evidently a matter of opinion and not of fact. There surely can

be no error in doing so; hence I agree to disagree with them. Until they can suggest a still better system I claim the right to hold to my opinion. Unless they recognize this division into two classes, they cannot of course agree with some of the deductions in the paper which are based on this division.

H. D. HIBBARD[5]: One might further urge that the word reduction be used in metallurgical language solely in its chemical meaning, not as is common, as a synonym for decrease, diminish, lessen, cut down, and others of similar portent. Occasionally it can only be told by the context if at all, whether the work is intended to mean decrease (as elimination by oxidation) or chemical reduction. If used in metallurgy and chemistry only in its chemical sense, such ambiguity would not occur.

CARL HERING (*Communicated*): Mr. Bancroft accuses me of not familiarizing myself with the literature; his logic is bad, since it is *because* I looked up the literature on some of these subjects and found such looseness of expressions, such differences of opinion, such inconsistencies, and in general such a lack of agreement and a lack of a rational system of terms, and such a "sloppy" use of terms, in chemistry, that I ventured to suggest these reforms. If Dr. Bancroft will familiarize himself with existing literature on such definitions and terms he will find my grounds for desirable reforms to exist. The very fact that this subject has been hotly discussed for years, is the best kind of proof that reforms are needed.

In abstracting my paper at the meeting, I called attention to the fact that hydrogen may perhaps have negative bonds, as in the metallic hydrides, for instance.

To have discussed the many details which Dr. Bancroft accuses me of not discussing, would have made the paper so long that few would have read it; I naturally had to limit myself to mere brief suggestions. Dr. Bancroft objects to recognizing a distinction between two different classes of bonds. This is a matter of opinion; we at least agree to disagree.

There was no misunderstanding about that "extraordinary statement from an unnamed chemist"; this very statement was one of my reasons for suggesting reforms. I am pleased that Dr. Bancroft at least agrees with me that it was "extraordinary."

[5] Consulting Metallurgist, Plainfield, N. J.

The question of whether it is a "simplification" to use a consistent and rational system of terms and definitions instead of using an inconsistent and irrational one, can I think be best left to the student or beginner rather than to the expert who is so familiar with these inconsistencies that they no longer trouble him to understand. There is a wise proverb that an old dog does not like to be taught new tricks. Electrical engineers have such a simple, rational and consistent system of units, terms and definitions, that they are more likely to notice the need of it in the science of chemistry than the expert chemists are. It is noteworthy that Dr. Bancroft has not pointed out any actual errors in my arguments; a wholesale condemnation of a paper without the support of sufficient and good reasons, can carry no weight with the unbiased, and is not proper and useful discussion.

Concerning the discussion by Prof. Richards, the question of whether it is desirable or not that we consider the faraday as a "practical unit" in terms of which those impracticable small electrons may be correctly expressed and measured, is a matter of opinion. When the theoretical units are impracticably small or large, and especially when their values are not known definitely, it is very desirable in practice to use a so-called "practical unit," especially when its value can be very definitely established. The volt and ohm, in place of the impracticable C. G. S. units, are good illustrations.

I do not think Prof. Richards or any one else can prove that it is a "fundamental error" for me to suggest that free charges and the corresponding bonds are alternatives of each other, and that each may be convertible into the other, or replace the other, hence that either but not both may exist. If this is true, and if chemical reduction is a reduction of valence, then it seems to follow as a necessary consequence that the real physical act of reduction must take place when these bonds are reduced in number by being converted into free charges. I admit that this is not the orthodox way of considering it, but until there exists some physical proof that it is wrong, it cannot be called an error, as the argument or deduction has not been shown to be irrational.

Answering his question about the reduction of ferric sulphate by solution, I believe this may not yet be known, but to be consistent it must be supposed to be reduced by the number of bonds

which correspond to the number of + charges which chemists place over the Fe atom in their formulas for ionized ferric sulphate, and which replace the bonds. He should ask the chemists how they know how many of these signs to use.

Quoting a part only of one of my sentences and omitting an essential part, and then ridiculing it, is surely not proper discussion. If we were as sure of these thermal relations as we are that the moon is not made of green cheese, I would not have wasted time to discuss them. In ancient times men were punished even by death for claiming that it was more logical and consistent to believe the earth to be round.

A physical law in which the constant is the same for *all* the elements should surely be considered to be a more fundamental one than another in which the constant is different for each element. In their gaseous form the different elements are more nearly alike physically than in their solid form, hence a law based on the gaseous state eliminates some of the differences between them and should therefore be considered the more fundamental one.

I should say that a "careful chemist" also prefers consistency and clearness in his terms and definitions, which surely cannot be claimed to be the case for some of the chemical terms. I agree to disagree with Prof. Richards on what constitutes a "careful chemist."

Prof. Frary's logic is faulty. If he will read his own quotation again he will find that the reason I gave for believing that unorthodox statement to be probably correct is because it "brings about a logical consistency which did not exist before," in other words it makes things fit which did not fit before. Incidentally, things that fit and are consistent are simpler and easier for the student to understand.

I suggested the term "electrolysis" myself, for the discharging process.

Concerning zero valence, Prof. Frary does not recognize that it would simplify matters if bonds were divided into two classes having different properties. Many chemists have already adopted the conception of zero valence; Prof. Frary is simply not one of them. I maintain that his students would have obtained a clearer

insight of the subject had he adopted this conception, as Prof. Weber has done.

A number of the reforms suggested in my paper were not new with me but were merely endorsed in the paper; I had supposed that this was known to chemists. In those cases Prof. Bancroft differs from the original authors, who, like myself, are also chemists. In this connection I take pleasure in referring to the pioneer work of the late Otis C. Johnson in the earlier editions of Douglas & Prescott's Manual, and to a paper by C. J. Reed in the Transactions of the St. Louis Academy of Science, 1886. Zero and negative valences are also described (and endorsed) in other books and papers, as is also the question of whether reduction is a loss or a gain of negative electrons, all of which I had supposed was known.

A paper presented at the Thirty-second General Meeting of the American Electrochemical Society, in Pittsburgh, October 5, 1917, President Fink in the Chair.

THE POISONING OF CATALYTIC AGENTS

By Wilder D. Bancroft.*

[Abstract.]

Commencing with the historic case of the poisoning of the platinum catalyzer by arsenic, in the contact sulphuric acid process, the writer discusses other catalytic processes in which similar actions take place, such as the Deacon chlorine process, Haber ammonia process, the Sabatier hydrogenization of oils, etc. The reasons are discussed, prominent writers on the subject being quoted at length, from Faraday to the present. The question of adsorption of gases by the catalyst is brought up and discussed, particularly in the case of charcoal as a catalyst. The effect of the reaction products in cutting down the speed of the reaction, and thus apparently poisoning the catalyst, is considered. The intimate relation between adsorption and catalytic action is emphasized. The application of these ideas to the phenomena of over-voltage is suggested, considering that the electrodes may act as catalyzers for the transformation of the liberated gases from the atomic into the molecular state.

One of the striking things about catalysis by solids is the way in which the catalytic action of the solid may be reduced enormously by the presence of relatively small amounts of certain substances which are therefore said to poison the catalytic agents. In the contact sulphuric acid process,[1] for instance, a stage was soon reached in the laboratory where the reaction worked well with purified gases and there was no noticeable deterioration of the contact material. When the experiments were repeated on a commercial scale, the contact material soon became inert. It was

* Professor of Chemistry, Cornell University.

[1] Knietsch: Ber. deutsch. chem. Ges., 34, 4069 (1901).

found that this disastrous result was due to impurities in the burner gases. Even when present in apparently negligible amounts, these impurities play havoc with the catalytic action of the platinum. Arsenic is the most dangerous impurity, and next comes mercury, while antimony, lead, iron, zinc, etc., act only in so far as they settle on and actually blanket the contact material. An amount of arsenic equal to ½ percent of the platinum in the contact material will destroy the catalytic action completely. In view of the enormous volumes of burner gas which are handled, it is easy to see to what an infinitesimal value the arsenic content must be reduced.

In the Deacon process for making chlorine from hydrochloric acid and oxygen, cupric chloride is the catalytic agent. This catalytic agent is very sensitive to the effect of impurities, such as sulphur dioxide, sulphur trioxide, and arsenic. It is because of this sensitiveness to sulphur dioxide that the hydrochloric acid from the Hargreaves salt-cake process cannot be used in the Deacon process. Water is also bad for the reaction, presumably because it is one of the decomposition products.[2]

The Haber ammonia process is very susceptible to poisons. Among the harmful substances are sulphur, selenium, tellurium, phosphorus, arsenic, boron, and their compounds; many carbon compounds; lead, zinc, bismuth, tin, etc.[3] Such minute quantities of the above-mentioned substances as are to be found almost always in the present commercial products and so-called pure gases are sufficient to diminish seriously the catalytic efficiency. For instance, an impurity of 0.01 percent sulphur in iron renders it nearly useless.

"In view of these facts, every care is taken to obtain pure contact material and to free the reacting gases from all poisonous bodies. The first is a comparatively easy matter, the ordinary methods of purification of course being employed. The continuous removal of contact poisons from the reacting gases is a more difficult undertaking, particularly as chemical contingencies have brought about the replacement of the electrolytic hydrogen employed by the impure hydrogen obtained from coal. This latter method of production depends upon the separation by liquefaction

[2] Bancroft: Jour. Phys. Chem., **21**, 573 (1917).

[3] Jobling: Catalysis and Its Industrial Applications, 44 (1916).

of the constituents of water-gas, hydrogen and carbon monoxide. Traces of carbon monoxide have then to be eliminated, together with the gaseous impurities derived from the decomposition of the coal, as well as the contaminations arising from the use of piping, lubricating oil in the pumps, etc. A development of this process of hydrogen production has recently been patented, in which carbon monoxide and steam are caused to interact under pressure at 300°-600°, in the presence of a catalyst such as iron, nickel, and the like, thereby producing carbon dioxide and hydrogen, of which the former is removed by absorption, leaving hydrogen ready compressed for the ammonia synthesis. The nitrogen is usually obtained by the fractionation of liquid air.

"In general the gases are filtered, washed, and then conducted over various solid absorption agents, whilst in some cases further purification is effected by passage over a portion of the same material as is employed as catalyst, at a raised temperature, before introduction into the catalyzer. This material is renewed repeatedly. The yield is found to be increased if both gases are deprived of water or of substances capable of producing water."

It is very difficult to get any definite facts in regard to the Ostwald nitric acid process. According to Jobling[4] "crude gas liquor formed the source of the ammonia first employed, the liquor being brought into contact with the hot air on the counter-current principle. The resulting mixture was purified by washing, and passage over milk of lime to remove carbon dioxide and sulphuretted hydrogen. In some cases no purification was effected, the products of the combustion of sulphur compounds being easily removed from the nitric acid by a single operation, while those coming from nitrogen compounds, such as aniline, pyridine, and prussic acid themselves form nitric acid."

This statement seems very improbable, there being some who say that ammonia from by-product coke ovens cannot be used at all and that the Ostwald process became successful only when pure ammonia was used, such as that from the Haber ammonia process or that obtained by the action of steam on calcium cyanamide, $CaCN_2 + 3H_2O = CaCO_3 + 2NH_3$. As a matter of fact, it is apparently not a very difficult matter to purify ammonia from the by-product coke ovens sufficiently to make it suitable for the

[4] Catalysis and Its Industrial Applications, 47 (1916).

Ostwald process. I have not been able to find any statement whether air is used because it is cheaper or because the large excess of nitrogen helps to check the oxidation of ammonia to nitrogen.

In the Sabatier process it is indispensable to success[5] that "the materials, whether hydrogen, metal, or organic compound, must be as pure as can be obtained, for any impurity is readily adsorbed by the catalyst to the detriment of its activity. Sulphur and its volatile compounds, as well as the halogens[6] are particularly to be avoided in this respect, and, to a lesser degree, arsenic and phosphorus with their volatile compounds. Electrolytic hydrogen, or hydrogen of equivalent purity, appears to be essential. Even then the catalyst must, sooner or later, succumb to the toxic effects of minute impurities impossible to remove; in consequence, the desirability of frequent renewal needs emphasis."

The platinum catalysis of hydrogen peroxide solutions is poisoned by very many things[7]—hydrocyanic acid, iodine dissolved in potassium iodide, mercuric chloride, hydrogen sulphide, mercuric cyanide, carbon monoxide, hydroxylamine hydrochloride, aniline, hydrochloric acid, etc.

Since one or more of the reacting substances must be adsorbed by the solid catalytic agent for catalysis to take place, the poisoning must be due to decreased adsorption. We shall have an apparent poisoning of the catalytic agent if we have formed on it a solid, liquid, or gaseous film which cuts down the adsorption of the reacting substances. Berliner[8] has described some experiments on the adsorption of hydrogen by palladium foil which bring out clearly the enormous effect due to very slight amounts of impurity. "A piece of palladium foil, weighing 44.5 mg., which had not been ignited or purified on the surface by mechanical treatment, adsorbed no hydrogen in the course of several hours. When the metal was treated in a vacuum, 450 cubic millimeters of gas were set free, 114 times the volume of the palladium. After the apparatus had stood for two hours, hydrogen was let in but no perceptible adsorption took place. When the foil was removed from the apparatus, careful observation showed that the surface

[5] Jobling: Catalysis and Its Industrial Applications, 69 (1916.)

[6] [The nickel oxide must not be prepared from the chloride because of the great difficulty of washing out the last traces of adsorbed chloride.]

[7] Bredig: Zeit. phys. Chem., 31, 324 (1899).

[8] Wied. Ann., 35, 803 (1888).

was not clean, as was shown by the fact that water did not wet the palladium. It seems probable that the trouble was due to vapors of fats in the apparatus or to fat carried to the metal by capillary action. To test this, the foil was ignited in the blast lamp; when cold it was wetted at once by water. After standing one hour in the apparatus, the surface was so coated with a film of fat that the foil was not wetted by water any more. To prove that the surface impurities kept the metal from adsorbing hydrogen, the palladium was cleaned by being made the positive electrode in sulphuric acid. After being washed with water and dried between filter paper, the metal was put into the apparatus as quickly as possible and hydrogen let in at once. The palladium adsorbed 857 times its volume of hydrogen almost instantaneously. The metal can also be purified by igniting in the blast lamp. The foil was taken out of the apparatus and ignited in the blast lamp. If put back quickly into the apparatus and hydrogen let in at once, the metal adsorbed 860 times its volume of hydrogen. If there was a delay of an hour or two before the hydrogen was let into the apparatus, no adsorption took place. Similar results were obtained with platinum."

Berliner called attention to the importance of these results as affecting the catalytic action of metals; but his work does not seem to have made much impression in spite of the fact that the platinum used in making contact sulphuric acid is readily poisoned by minute quantities of arsenic, etc. The quantitative part of Berliner's work on the adsorption of hydrogen and oxygen has been criticized severely by Ramsay, Mond and Shields[9] because of the very small volumes of gas with which Berliner worked.

Bodenstein[10] found that traces of fat cut down the rate at which platinum accelerates the formation of water from oxyhydrogen gas. The most intense action occurs only when the platinum surface is very clean, and can be obtained in a reproducible manner only when the purity of the surface is a maximum, a state of things which is very difficult to obtain and almost impossible to maintain. "The purification of the platinum was done at first by heating with concentrated sulphuric acid or with a mixture of nitric acid and sulphuric acid, later by treating with a mixture of

[9] Zeit. phys. Chem., 19, 58 (1896).
[10] *Ibid.*, 46, 730 (1903).

chromic and sulphuric acids followed by washing with freshly distilled water, which was protected as much as possible from contamination by the ordinary laboratory gases. The platinum was placed in the apparatus while still moist and was then brought in contact with the gases which were either not dried at all or were dried slightly by passing over calcium chloride. After the first difficulties had been overcome, reproducible results could almost always be obtained in this way.

"In order that the effectiveness of the platinum might be kept constant, all the other parts of the apparatus were of course kept scrupulously clean. The large joints, including one with 5 cm. opening through which the platinum ball was at first introduced, were ground so carefully that when moist they closed absolutely gas-tight and therefore never needed to be greased. Vapor from the mercury in the manometer could not be kept out, however, and the stop-cock on the gasometer could not be made tight without the use of grease. I tried, of course, to use glassy phosphoric acid as lubricating medium; but it becomes diluted too rapidly in the moist gases and then the joint is not tight any more. Consequently the effectiveness of the platinum always decreased gradually, though so slowly in the successful experiments that the results are comparable. In the experiments with an excess of oxygen, the catalytic action of the platinum once remained constant for several days. This looks as though the chief disturbing factor were the grease from the stop-cock, which was oxidized in this case."

It has been pretty well understood for eighty years or more that impurities cut down the catalytic action of platinum. Faraday[11] is very clear on this point. While studying his recently invented voltameter, he found that the platinum electrodes, if very clean, caused the oxyhydrogen gas to react after the current was cut off. The platinum could be cleaned electrolytically, mechanically, or by heat. The platinum would not work indefinitely; but Faraday pointed out that it retained its efficiency longer the purer the hydrogen and oxygen. Faraday pointed out that platinum which had ceased to be active was no longer wetted readily by water, thus showing the presence of some sort of greasy film. While this could be readily accounted for, Faraday was very much puz-

[11] Experimental Researches on Electricity, 1, 165 (1839).

zled by the fact that gases like carbon monoxide or ethylene had a very marked effect in cutting down the catalytic action of the platinum. Faraday was not able to account for this at all. He says: "Whether the effect produced by such small quantities of certain gases depends upon any direct action which they may exert upon the particles of oxygen and hydrogen, by which the latter are rendered less inclined to combine, or whether it depends upon their modifying the action of the plate temporarily (for they produce no real change on it), by investing it through the agency of a stronger attraction than that of the hydrogen, or otherwise, remains to be decided by more extended experiments."

Turner[12] was on the right track when he said that "one is tempted to suppose that these gases act by soiling the metallic surface, though in some respects this is not satisfactory." This explanation was premature, however, and a different one was offered later by Henry[13] who amplified Faraday's experiments, correcting some of his results. Henry found that adding carbon monoxide to a mixture of hydrogen and oxygen in presence of platinum does not actually prevent the reaction between hydrogen and oxygen, but retards it, so that the reaction takes place very slowly and most of the oxygen reacts with the carbon monoxide. Starting with the mixture $2CO + 2H_2 + O_2$ Henry found that about ninety percent of the oxygen reacted with the carbon monoxide and only about ten percent with the hydrogen. When the ratio of hydrogen to carbon monoxide was increased in the original mixture less of the oxygen reacted with the carbon monoxide. Faraday had found that a mixture of carbon monoxide and oxygen did not react appreciably in several days when in contact with a clean platinum plate, but Henry found that a reaction does take place in presence of platinum foil, platinum sponge, and platinum black. The reaction is very slow with platinum foil, more rapid with platinum sponge and fastest of all with platinum black. Since hydrogen and oxygen react much more rapidly in presence of platinum than do carbon monoxide and oxygen, one would naturally expect preferential burning of hydrogen from a mixture of hydrogen and carbon monoxide, whereas one actually gets preferential burning of carbon monoxide. Henry recog-

[12] Chemistry, 5th ed., 647.
[13] Phil. Mag. (3), 9, 324 (1836).

nized this difficulty and met it in a very clever way, though his argument is not absolutely sound.

"An obvious objection, however, presents itself to the view that carbonic oxide possesses a stronger affinity for oxygen than hydrogen exerts, *viz.*, that while hydrogen and oxygen are speedily detonated at common temperatures by the plate or sponge, the union of carbonic oxide and oxygen takes place with great slowness. The explanation of this apparent anomaly, I believe to be, that the product of the combustion of hydrogen (aqueous vapor) at once quits the surface of the metal and is liquefied by the cold sides of the tube; while the combustion of the carbonic oxide yields a gas which remains for a while adherent to the metallic surface next to which it is generated, and thereby prevents a sufficiently rapid access of fresh unaltered gas to elevate materially the temperature of the platina. In confirmation of this view I found that caustic potassa, by absorbing the carbonic acid as it is formed, accelerated the acidification of carbonic oxide. When the metallic superficies is so extensive (for instance, in the powder of Liebig) that a high temperature is attained quickly by the metal in contact with the first portions of gas that are consumed, it has been shown already that carbonic oxide, like hydrogen, unites with oxygen with incandescence. Finally, it is well known that even mixtures of hydrogen and oxygen do not detonate on first admitting the prepared plate. During the first minute the union is generally very slow, and it only becomes explosive when the temperature of the plate has been raised by its action upon the gaseous mixture."

Although the reaction between carbon monoxide and oxygen is practically irreversible at ordinary temperatures, Henry recognized that the presence of the reaction product might slow up the reaction and he proved it by removing the carbon dioxide with caustic potash, thereby increasing the reaction velocity. That was very clever of him, because most people nowadays would have been so hampered by their incomplete theoretical knowledge of reaction velocities that they would have been certain that the reaction products could not affect the reaction velocity. Henry's argument is unsound, however, in several respects. In the first place he has not shown that carbon monoxide and oxygen react more rapidly than hydrogen and oxygen when the carbon dioxide

is removed continuously. He has shown that there is an increase in reaction velocity, but not that the increase is sufficient to account for the phenomena. In the second place the rate of oxidation of the carbon monoxide cannot be the determining factor because Henry obtained preferential burning of carbon monoxide in the absence of alkali. What happens is that the adsorption of carbon monoxide by platinum cuts down the adsorption of hydrogen to such an extent that the reaction between hydrogen and oxygen becomes almost negligible.

This is not an especially surprising result. Freundlich[14] points out that the general rule for mixtures of gases seems to be that the more readily adsorbed gas displaces the other to some extent and is adsorbed to a greater relative amount than one would have predicted from experiments on the single gases. This cannot be anything more than a first approximation, because with readily condensible gases one of the vapors may help carry down the other. Cases of this sort have been studied by Hunter,[15] the most striking instance being water and ammonia. It is probable that the adsorbed water dissolves or adsorbs ammonia, thus increasing the amount of ammonia apparently held by the charcoal. Displacements also occur when two solutes may be adsorbed from solution. Schmidt[16] has studied the simultaneous adsorption of iodine and acetic acid by charcoal from solutions in water and in ethyl acetate. With both solvents less of each substance was adsorbed than if the other had not been present. Schmidt generalizes to the effect that "when charcoal is in contact with a solution containing the two solutes A and B it adsorbs less of each than when it is treated with the same substances A and B singly. If charcoal is in equilibrium with any substance A dissolved in any solvent, the addition of a second substance B will force some of A out of the charcoal and *vice versa*. The total amount which is adsorbed when two solutes are present simultaneously in solution is always less than the arithmetical sum of the amounts of these substances that would be adsorbed singly."

This generalization is undoubtedly too broad. A more careful study will show that the special properties of the substances A and

[14] Kapillarchemie, 99 (1909).
[15] Jour. Chem. Soc., 23, 73 (1870).
[16] Zeit. phys. Chem., 74, 730 (1910).

B will have to be taken into account and that we shall find cases where the total adsorption will be greater than the sum of the single adsorptions, though no such cases seem as yet to be on record. Lake[17] has shown that one acid dye does not apparently displace another on wool or silk; but of course the absolute concentrations are very small in these cases. Freundlich and Masius[18] studied the adsorption of pairs of organic acid and obtained results similar to those of Schmidt. They also found that the acid, which is adsorbed the more, is displaced the less when the two acids are present in the solution. These experiments of Schmidt, and of Freundlich and Masius, throw light on Skey's results with nitric and sulphuric acids in presence of charcoal. Skey[19] found that small amounts of nitric acid were adsorbed completely from dilute solutions of sulphuric acid, but not from concentrated ones. With increasing relative concentrations of sulphuric acid, we should expect an increasing displacement of nitric acid from the charcoal, which is what actually occurred.

Rona and Michaelis[20] have studied the effect of acetone and acetic acid on the adsorption of grape sugar by charcoal. When 100 c.c. of a 1.04 percent sugar solution was shaken with 5 grams charcoal, the filtrate contained only 0.53 percent sugar. If the sugar solution contained 0.3, 3.0 or 15.0 c.c. acetone, the percentages of sugar in the filtrate were 0.64, 0.97, and 1.06 respectively. If 0.2, 1.0 or 10 c.c. concentrated acetic acid is added instead of acetone, the percentages of sugar are 0.60, 0.70, and 1.06 respectively. The difference between 1.04 and 1.06 would probably disappear if one corrected for the effect of acetone and acetic acid upon the optical rotation of sugar. In this case a moderate addition of acetone or acetic acid cuts the adsorption of sugar down to zero. On the other hand, Rona and Michaelis found that albumin and acetone were each adsorbed by charcoal as though the other were not there.

Lachs and Michaelis[21] found that caustic potash decreases very much the adsorption of potassium chloride from water by charcoal. In other words the presence of the strongly adsorbed hydroxyl ion

[17] Jour. Phys. Chem., **20**, 761 (1917).
[18] Van Bemmelen Gedenkboek, 88 (1910).
[19] Chem. News, 17, 217 (1886).
[20] Biochem. Zeit., **16**, 489 (1909).
[21] Zeit. Elektrochemie, **17**, 1 (1911).

decreases the adsorption of the chlorine ion very much. They also find that the adsorption is increased markedly if sulphuric acid be added in small amounts to a potassium chloride solution. This is in accordance with the results of Osaka[22] that potassium chloride is adsorbed more strongly than potassium sulphate, sodium chloride than sodium sulphate, and presumably hydrochloric acid than sulphuric acid. We therefore get hydrochloric acid adsorbed and potassium sulphate left in solution.

I do not know of any record of experiments on the adsorption of mixtures of hydrogen and carbon monoxide by platinum; but I feel sure that carbon monoxide will be found to cut down the adsorption of hydrogen very markedly. It is to be hoped that somebody will try this experimentally.

Faraday found that ethylene decreased the catalytic action of platinum on hydrogen and oxygen more than did carbon monoxide; but this result is not confirmed by Henry[23] and probably was due to some impurity in Faraday's ethylene. "Olefiant gas, even when constituting one-fourth or one-third of the mixture, did not in the slightest degree retard the action of the platina balls or sponge. Several trials were made, in which the olefiant and explosive mixture were mingled in equal proportions. In all of these the ball acted instantaneously, and ascended rapidly in the tube during one or two minutes, when its rise was suddenly checked. This 1.02 cu. in. were reduced in the first minute to 0.90, and became 0.82 after an hour's contact. The following day only 0.56 were left, which were not diminished by washing with potassa. Even when the volume of the olefiant was double that of the explosion mixture, there was an instant action, though to less extent than in the last experiment. When the volume of the olefiant was three times that of the explosive mixture, no immediate action was visible, though a notable diminution always took place in the course of a few hours or on the following day. Finally, the activity of the black powder of Liebig was not suspended by the addition of twenty volumes of olefiant to one of explosive mixture.

"On subjecting to examination the gaseous products of these and other experiments on olefiant gas, mingled with various pro-

[22] Mem. Coll. Sci. Kyoto, **1**, 267 (1915).
[23] Phil. Mag. (3), **65**, 329 (1836).

portions of the explosive mixture, very different results from those afforded with carbonic oxide were obtained. In the greater number of experiments with olefiant gas, no appreciable diminution was caused by prolonged contact, or by subsequent washing with potassa; and though in some cases, when the explosive mixture considerably exceeded the olefiant there was a perceptible absorption, yet the carbonic acid thus evidenced was always of small amount. In three successive experiments, in which olefiant gas and explosive mixture were mingled in equal proportions, there was no measurable product of carbonic acid. Yet in all these cases, though the ball ascended rapidly on first contact, its activity was soon suspended. Olefiant gas then possesses, like carbonic oxide, an undoubted power of retarding the union of hydrogen and oxygen, but it differs from carbonic oxide in not necessarily affording carbonic acid by so acting.

"Similar differences presented themselves on submitting mixtures of olefiant and oxygen only to the action of platina. To the observation of Dr. Faraday, 'that the most prolonged contact with the prepared plates never induced the union of the elements of olefiant with oxygen,' I may add, that the contact was not efficient, even when aided by the presence of liquid potassa. The spongy metal, similarly conjoined with potassa, was also for the most part entirely inert, and in a few instances in which carbonic acid appeared, it was formed very slowly, and in small quantity. At the temperature 480° F., however, the sponge has been shown to occasion the speedy though inexplosive combustion of olefiant in mixture with oxygen. Liebig's powder too caused the slow combination of the two gases at atmospheric temperatures, as was evidenced both by considerable diminution of volume, and by the test of lime water; and when the tube containing the gaseous mixture and powder was surrounded with boiling water, combination ensued with rapidity. Now the mode in which platina acts is of course identical in the various states of plate, sponge, or powder; the powder presenting only an infinitely larger extent of surface for gaseous contact than the plate or sponge. Hence the tendencies of the constituents of olefiant to unite with oxygen, which are evidenced by combinations to considerable amount in presence of the powder, may be inferred to be operative, though less effectively, on the surface of the same metal under other forms; and

viewed in connection with the unequivocal proofs of the nature of the interference of carbonic oxide, these tendencies may be admitted to furnish an adequate explanation of the more feeble interference of olefiant gas. The interfering power of carbonic oxide as respects the action of the platina balls is eighteen times as great as that of olefiant; carbonic oxide in the proportion of 1/24th interfering as completely as olefiant in that of ¾ths of the mixture.

"The influence of temperature in quickening the action of the sponge or powder on mixtures of olefiant and oxygen strongly confirms this view, when taken in connection with a peculiarity observed in the mode of interference of olefiant gas. Thus it has been stated that olefiant mingled in equal bulk with explosive mixture did not prevent instant action; and that it began to interfere only when much of the oxygen and hydrogen had combined together, with the disengagement of great heat. The interfering power of olefiant gas, feeble at atmospheric temperatures, is then greatly augmented by heat, which had been already shown to determine the separate combination of the elements of olefiant with oxygen, and which is well known to exalt chemical affinity.

"In the recapitulation, it may be stated that carbonic oxide interferes with the action of platina upon mixtures of oxygen and hydrogen, by virtue of its stronger affinity for oxygen, which causes it slowly to take the larger portion of that gas. Olefiant gas, which at common temperatures has a weaker attraction for oxygen than hydrogen, suspends the combining tendencies of those two gases, only when its volume greatly exceeds that of the mixture, in which case the weaker affinity is aided by a greater number of atoms. Even with this advantage olefiant is unable to appropriate the oxygen to itself, but only retards its union with hydrogen by opposing a weaker attractive tendency."

The relatively slight retarding action of ethylene is quite in accord with its presumably being adsorbed less strongly than hydrogen by platinum. If it only cuts down the adsorption of hydrogen appreciably when present in relatively large excess, it will have very little effect on the rate of combustion of oxyhydrogen mixtures. It is not possible to get satisfactory data as to the way in which platinum adsorbs mixtures of hydrogen and ethylene, because platinum causes these two gases to react.[24] It is not

[24] Harbeck and Lunge: Zeit. anorg. Chem., 16, 34 (1898).

known to what extent this is a disturbing factor in Henry's experiments. It is quite natural under the circumstances that Henry should have drawn the conclusion that "the property inherent in certain gases of preventing or retarding the union of hydrogen and oxygen is to be referred to their attraction for oxygen and not to any peculiar action of the metallic surface by which it becomes invested with the interfering gas." That this is wrong can be seen from the fact that carbon monoxide is at least equally effective in checking the very different reaction between hydrogen and ethylene in presence of platinum. Here there is no oxygen and no oxidation and consequently the attraction of carbon monoxide for oxygen can play no part. The adsorption hypothesis explains both reactions equally well. If carbon monoxide cuts down the adsorption of hydrogen by platinum, it will interfere with the reaction between hydrogen and oxygen or ethylene. Henry's argument in regard to the respective combustibilities of hydrogen, carbon monoxide, ethylene and methane, holds only for certain conditions because under others methane and ethylene are burned more readily than either hydrogen or carbon monoxide.

Schönbein[25] pointed out that the hydrides of sulphur, selenium, phosphorus, arsenic, antimony, and tellurium act very energetically in cutting down the catalytic action of platinum on mixtures of air with hydrogen or ether. He is quite clear that the trouble is due to the formation of some sort of a film over the platinum and he considers the possibility that we may be dealing with a gas film of hydrogen sulphide, phosphide, etc. Schönbein rejects the assumption of a gas film on the ground that hydrogen sulphide, for instance, is more readily oxidized by oxygen than hydrogen, and that consequently such a film would be destroyed at once. He therefore decides that the film is probably solid sulphur, phosphorus, selenium, arsenic, antimony or tellurium, though he admits that this should be checked experimentally. I do not know whether Schönbein is right or not; but his reasoning is not necessarily sound. We have already seen that the combustion of mixed gases may proceed along very different lines in the presence and in the absence of a catalytic agent. It might be that hydrogen sulphide was adsorbed by platinum so much more strongly than its oxidation products that the platinum

[25] Jour. prakt. Chem., 29, 238 (1843).

actually stabilized the hydrogen sulphide. This is a question which should not be difficult to determine experimentally. The really important thing is that Schönbein saw so clearly that one must postulate the existence of a film of solid, liquid, or gas over the surface of the platinum if one is to account intelligently for the poisoning of the platinum catalysis.

So far, all that I have done is to show that we can account for all the cases that have been considered if we postulate suitable and not improbable adsorptions. To make the case complete one ought to show that all substances which poison catalytic agents do cut down the adsorption of the essential reacting substance or reaction product.

One case involving a gas has been studied. Harbeck and Lunge[26] found that carbon monoxide prevents the catalytic action of platinum black or palladium on a mixture of ethylene and hydrogen. Special experiments showed that both platinum and palladium take up carbon monoxide and retain it pertinaciously. Lunge and Harbeck say that "both metals form compounds with carbon monoxide which are so stable that they must be considered as definite chemical compounds even though it is not possible to convert all the platinum or palladium into the corresponding compounds and to obtain them in a pure state for analysis. A purely mechanical adsorption of carbon monoxide is not probable because the carbon monoxide cannot be driven out by treatment with hydrogen or other gases. Also after previous treatment with hydrogen the platinum takes up just as much carbon monoxide as without the treatment with hydrogen. Also it is only by assuming the existence of an actual chemical compound between Pt and CO that one can account for the fact that the contact action of platinum on a mixture of ethylene and hydrogen is checked so completely.

"The carbon monoxide is not set free until the platinum is heated to about 250° and then it comes off suddenly, which is also an argument for the existence of an actual chemical compound. These compounds of platinum and palladium are like those of carbon monoxide with nickel and iron. Since they are not volatile nor soluble in the ordinary solvents, it is not possible to isolate them or to decide upon their compositions."

[26] Zeit. anorg. Chem., 16, 50 (1898).

This was the proper view to take at the time when these experiments were made; but, nowadays, it is clear that there is no real evidence of the existence of a definite compound. If the hypothetical compound is as stable as Lunge says, there should be no difficulty in obtaining the compound in a pure state. If this cannot be done—and Lunge's experiments are not conclusive on this point—we must be considering a case of adsorption. In any event the cutting down of the catalytic action is due to the fact that the ethylene and hydrogen are not adsorbed to the same extent as when no carbon monoxide is present. Except from a theoretical point of view, it is immaterial whether the platinum is coated with a film of compound or with a film of adsorbed carbon monoxide.

Freundlich and Kaempfer[27] have studied an interesting case of the cutting down of adsorption in aqueous solution. Uranium X_1 is adsorbed strongly by charcoal and is displaced to some extent by very dilute solutions of thorium nitrate. The presence of a solution containing about 0.0004 millimol $Th(NO_3)_4$ (a little less than 0.2 mg. per liter) increases the concentration of uranium X_1 in the solution by about twenty percent. The charcoal is thus poisoned, so far as the adsorption of uranium X_1 is concerned, by infinitesimal amounts of thorium nitrate. When a stronger solution of thorium nitrate was used, the concentration of the uranium X_1 in the solution increased to over five-fold. It has been shown by Warburg[27a] that the oxidation of oxalic acid in presence of a suspension of blood charcoal in water is checked by methyl urethane, ethyl urethane, propyl urethane, and phenyl urethane, in this order, the phenyl compound being the most effective. It was recognized that this was due to the urethanes cutting down the adsorption[27b] of oxalic acid.

We have already seen that Henry increased the rate of reaction between carbon monoxide and oxygen in presence of platinum by adding caustic potash to remove the carbon dioxide. It is easy to see that the piling up of the reaction products will cut down the reaction velocity if they prevent the reacting products from coming in contact with the catalytic agent. This has been ob-

[27] Zeit. phys. Chem., **90**, 681 (1915).
[27a] Pflüger's Archiv, **155**, 54 7(1914).
[27b] Höber: Physikalische Chemie der Zelle und Gewebe, 467 (1914).

served in the contact sulphuric acid process.[28] The usual explanation is that we are dealing with diffusion only; but it is more likely that there is a cutting down of the adsorption, since this is the most important factor when carbon monoxide poisons platinum with reference to the hydrogen-ethylene reaction. A special case of some interest is the reaction between carbon monoxide and oxygen at about 300° with quartz glass as catalytic agent.[29] "Oxygen acts at any rate qualitatively about as it should, the rate of reaction increasing with increasing concentration. Carbon monoxide behaves quite differently and tends to check its own combustion under these circumstances. It is a negative autocatalytic agent. In all the experiments the peculiar result was obtained, that the absolute reaction velocity increases with decreasing concentration of carbon monoxide and decreases with increasing concentration." Bodenstein offers no explanation for this surprising result though he calls attention to the fact that carbon monoxide acts as a poison in many cases. There are no data in regard to the adsorption of carbon monoxide by quartz glass from which one could predict the results obtained by Bodenstein and Ohlmer; but if carbon monoxide displaces oxygen sufficiently at the surface of the catalytic agent, one would get exactly the results that Bodenstein and Ohlmer found experimentally. It is therefore probable that the action of carbon monoxide in poisoning its own combustion with oxygen is due to its displacement of oxygen from the adsorption film. When discussing the fractional combustion of carbon monoxide and hydrogen in presence of platinum, stress was laid on the fact that carbon monoxide must cut down the adsorption of hydrogen by platinum. It is also possible that carbon monoxide cuts down the adsorption of oxygen by platinum though this cannot be the important factor because carbon monoxide also poisons the reaction between hydrogen and ethylene in presence of platinum.

A case which has been studied a great deal quantitatively is the decomposition of hydrogen peroxide solution by platinum. One of the most interesting things about this reaction is its extreme sensitiveness to the so-called poisons.[30] The rate of decom-

[28] Bodenstein: Zeit. Elektrochemie, 9, 696 (1903); Zeit. anorg. Chem., 44, 267 (1905).

[29] Bodenstein and Ohlmer: Zeit. phys. Chem., 53, 166 (1905).

[30] Bredig and von Berneck: Zeit. phys. Chem., 31, 258 (1899); Bredig and Ikeda: *Ibid*, 37, 1 (1901).

position of hydrogen peroxide by a given solution of colloidal platinum was reduced approximately to one-half by N/20,000,000 HCN, N/2,000,000 $HgCl_2$ and N/300,000 H_2S. Curiously enough it has been shown by Schönbein[31] that these same substances cut down the catalytic action of red blood corpuscles on hydrogen peroxide. Owing to the similarity between the action of colloidal metals and organic ferments in many cases Bredig called the col-

TABLE I.

Concentrations at which poisons reduce the rate of the catalytic decomposition of hydrogen peroxide approximately to one-half.

Poison	Colloidal Platinum	Haemase
H_2S	N/300,000	N/1,000,000
HCN	N/20,000.000	N/1,000,000
$HgCl_2$	N/2,000,000	N/2,000,000
$HgBr_2$	.....	N/300,000
$Hg(CN)_2$	N/200,000	N/300,000
I_2 in KI	N/5,000,000	N/50,000
$NH_2OH.HCl$	N/25,000	N/80,000
Phenyl hydrazine	.....	N/20,000.
Aniline	N/5,000	N/400
Arsenious acid	N/50	No poisoning at N/2,000
CO	Very poisonous	No poisoning
HCl	N/3,000	N/100,000
NH_4Cl	N/200	N/1,000
HNO_3	No poisoning	N/250,000
H_2SO_4	No poisoning	N/50,000
KNO_3	No poisoning	N/40,000 at 0°
$KClO_3$	Slight poisoning (?)	N/40,000 at 0°

loidal metals prepared by his method inorganic ferments. This stirred up a violent discussion because people seemed to think that the colloidal metals ought to behave exactly like the ferments in every respect or ought not to behave like them at all. In Table I are given data[32] for the concentration necessary to cut down the rate of decomposition of hydrogen peroxide to one-half when the catalytic agents are colloidal platinum and haemase, the active enzyme in the red blood corpuscles. While many of the substances that cut down the platinum catalysis also decrease the catalytic action

[31] Jour. prakt. Chem., **105**, 202 (1868).
[32] Senter: Zeit. phys. Chem., **51**, 701 (1905).

of haemase, there are many cases where no parallelism occurs. Thus carbon monoxide is very toxic to platinum and has no effect on haemase. On the other hand, nitric acid, sulphuric acid, and potassium chlorate have practically no effect on platinum and are quite toxic to haemase. Even where there is a general parallelism, one must not follow it too closely. With platinum the prussic acid solution has one-tenth the concentration of the mercuric chloride solution, while with haemase it has double the concentration. The tabulated concentration for mercuric chloride is the same for platinum as for haemase, while that for iodine dissolved in potassium iodide is one hundred times as great for haemase as for platinum. Kastle and Loevenhart[33] point out that prussic acid is a strong poison for colloidal platinum and colloidal silver, but accelerates the catalysis of hydrogen peroxide by iron and copper. The whole matter becomes a good deal clearer if we consider that the so-called poisons are adsorbed strongly by the catalytic agent and therefore prevent or decrease the adsorption of hydrogen peroxide.[34] This adsorption will be specific and will therefore in general not be the same quantitatively for different catalytic agents. On the other hand, there may well be a qualitative agreement in some or even in many cases, which is exactly what is found experimentally. Owing to the extreme flexibility of this hypothesis there is nothing as yet to prevent its accounting for all the phenomena observed.[35] On the other hand, it must be remembered that proof is still lacking that platinum adsorbs prussic acid, for instance, very much more strongly from an aqueous solution than it does hydrogen sulphide or that there is no such difference with haemase. It is also to be remembered that there is no experimental proof as yet that platinum adsorbs the poisons strongly.[36] We do know, however, that many of the substances that poison the platinum catalysis of hydrogen peroxide also poison the platinum catalysis of oxyhydrogen gas. In fact, Ernst[37] arranges the substances which he studied in the following order in regard to their poisoning action on the platinum catalysis of oxyhydrogen gas, the strongest poison

[33] Am. Chem. Jour., **29**, 397 (1903).
[34] *Cf.* Senter: Zeit. phys. Chem., **51**, 702 (1905); Denham: *Ibid*, **72**, 689 (1910).
[35] *Cf.* Freundlich and Kaempfer: Zeit. phys. Chem., **90**, 681 (1915).
[36] *Cf.* however, Meyerhof: Pflüger's Archiv, **157**, 251 (1914).
[37] Zeit. phys. Chem., **37**, 483 (1901).

coming first: Prussic acid, cyanogen iodide, alkaline sodium thiosulphate, mercuric chloride, hydrogen sulphide, iodine, bromine, phosphine, hydroxylamine hydrochloride, mercuric cyanide, hydrazine sulphate, arsenious acid, phenol. Ernst points out that this is also the order in which the substances poison the platinum catalysis of hydrogen peroxide. Although a ten percent solution of formic acid precipitates colloidal platinum, it increases the rate of reaction of oxyhydrogen gas three-fold. Ernst cites Ikeda as his authority for stating that formic acid has a similar effect on the platinum catalysis of hydrogen peroxide.

There may be other factors which must be taken into account. Thus Bredig[38] points out that when colloidal platinum is allowed to stand in contact with hydrogen peroxide and concentrated potassium cyanide, the platinum flocculates and precipitates. The agglomerated platinum causes the hydrogen peroxide to decompose, thus showing that the cyanide does not poison precipitated platinum black. There seem to be only two possible explanations. One is that the adsorption of potassium cyanide by platinum falls off very much more rapidly with increasing size of the platinum particles than does the adsorption of hydrogen peroxide by platinum. The other explanation is that, through oxidation or otherwise, there is formed what might be called an anti-body, which cuts down the adsorption of the cyanide. Neither hypothesis is very satisfactory and neither is supported by any experimental evidence that I know of except that Jablczynski[39] found that platinized platinum decomposes hydrogen peroxide one hundred times as fast and chromous chloride only three times as fast as does smooth platinum foil. The matter should be taken up because Rosnyak[40] claims that the catalytic action of platinum on hydrogen peroxide increases with increasing disperity while Felgate[41] states that pulverulent nickel reduces nitric oxide and colloidal nickel does not. In both cases it is probable that some factor has been overlooked or not stated. The more finely divided the platinum the more likely the surface is to be coated with oxide and it does not follow at all that the catalytic action of metallic platinum is the same as that of an oxide of platinum. If the metal

[38] Zeit. phys. Chem., **31**, 332 (1899).
[39] *Ibid.*, 64, 751 (1908).
[40] *Ibid.*, 68, 85 (1913).
[41] Chem. News, **108**, 178 (1913).

is kept in suspension by means of gelatine or some similar substance, this will have an effect on the adsorption and consequently on the catalytic agent. The increase in stability of the colloidal metal may have to be paid for in a decreased catalytic action.

As a result of the hypothesis developed in this paper it is now possible to offer a plausible explanation of some electrolytic phenomena which have been most puzzling hitherto.[42] "It is very remarkable that the rise of potential at a smooth platinum anode is sometimes dependent on the nature of the electrolyte. The difference of potential between smooth and platinized platinum is less in sulphuric acid than in caustic soda, other conditions being the same. A slight addition of hydrofluoric acid to sulphuric acid increases the potential at which oxygen is set free at smooth platinum by an amount varying from several hundredths of a volt to a tenth of a volt, provided the temperature is low and the current density not too high. The increase in potential is greater the ratio of hydrofluoric acid to sulphuric acid and reaches a maximum for pure hydrofluoric acid.[43] Hydrochloric and hydrobromic acids behave similarly except that with these acids the maximum potential is reached with slight additions so that the amount of the halide acids is only a few percent of the total acid concentration. Here also the potential in pure hydrochloric acid is higher than in an equivalent sulphuric acid solution in case a sufficient evolution of oxygen is provided by keeping the concentration of the hydrochloric acid sufficiently low and the current density sufficiently high. In normal HCl a current density of 0.057 amp./sq. cm. is sufficient.[44] Nitric acid is very variable. In normal acid solution it always raises the potential; but in a 10N. acid solution the addition of a small amount of acid raises the potential, whereas addition of more nitric acid lowers it a good deal, so much so that between 10N. H_2SO_4 and 10N. HNO_3 the potential for the evolution of oxygen[45] falls 0.28 to 0.65 volt for anode current densities of 0.05 to 0.5 amp./sq. cm. and a temperature of 12°. In caustic soda solution presence of NO_3 raises the anode potential. Ammonia has the same effect, because it is oxidized at the anode to nitrate.[46] * * *

[42] Foerster: Elektrochemie wässeriger Lösungen, 284, 735 (1915).
[43] Müller: Zeit. Elektrochemie, 10, 780 (1904).
[44] Müller and Scheller: Zeit. anorg. Chem., 48, 112 (1905).
[45] Schellhaass: Zeit. Elektrochemie, 14, 121 (1908).
[46] Müller and Spitzer: Zeit. Elektrochemie, 11, 920 (1905).

"It is very often customary to use platinum black as a means of accelerating the oxidizing action of atmospheric oxygen. Oxidations which take place readily at a platinized anode may therefore be considered as being accelerated catalytically by the platinum black of the anode. The power of platinum black to act catalytically in oxidations and in many other chemical reactions is affected very markedly by certain substances, such as prussic acid, mercuric cyanide, etc.[47] The same substances have a similar action on many electrolytic oxidations occurring at platinized anodes; they cut down the rate of oxidation and, when the current or the rate of oxidation is kept constant, cause an increase of the anode potential which gives a higher oxygen pressure in the solution and therefore restores the old reaction velocity. When a solution of sodium thiosulphate is kept absolutely neutral, it is oxidized quantitatively to tetrathionate[48] at a platinized anode with a potential of +0.44 volt for a current density of 3.10 to 4 amp./sq. cm. If one adds a trace of mercuric cyanide, the anode potential at which the same current is obtained rises to +0.48 volt. From this it follows that the oxidation process really takes place through an intermediate formation of oxygen according to the reaction

$$2S_2O_3'' + O + H_2O \rightarrow S_4O_6'' + 2OH'$$

and not, as one might have expected, according to the reaction

$$2S_2O_3'' + 2F \rightarrow S_4O_6''$$

In addition to these essentially catalytic actions of certain dissolved substances which are not used up during the electrolysis, there are some other remarkable phenomena which are distinctly different in nature. When discussing the anodic evolution of oxygen we became acquainted with the surprising and still unexplained fact that certain anions, such as Cl', F', SO''_4, ClO'_4, etc., increase markedly the anode potential at which oxygen is evolved at a smooth platinum anode. The presence of substances which increase the anode potential aids such anodic oxidations as call for

[47] Bredig and von Berneck: Phys. Chem., **31**, 324 (1899); Bredig and Ikeda: *Ibid*, **37**, 1 (1901).

[48] Thatcher: Zeit. phys. Chem., **47**, 693 (1904): It is still an open question to what an extent in these and similar cases the action of the retarding substances may or may not be due to the formation of thin diaphragms.

a high anode potential. Thus the current yield of persulphates is increased by the addition of small amounts of hydrochloric acid,[49] which increases the anode potential.[50] The oxidation of iodate to periodate does not take place in neutral solution at a smooth platinum anode, but takes place readily if the solution contains chloride, chlorate, or sulphate in addition to iodate.[51] The electrolysis of acetates at smooth platinum gives rise chiefly to ethane and carbon dioxide; but the products are chiefly methyl alcohol and carbon monoxide [?] when the acetate solution contains chlorate, sulphate or bicarbonate.[52]. Fluoride ions are especially active; they increase the oxidation of chromic sulphate to chromic acid or of manganous sulphate to permanganic acid.[53] In the electrolysis of potassium iodate, addition of fluorides causes an increase in the current yield of periodate from zero up to 30 percent;[54] in the electrolysis of potassium sulphate addition of fluorides increases the yield from 50 percent to 80 percent.[55] In these last cases the fluorine anions cause a marked increase in the anode potential. In the instances of electrolytic reduction there is nothing comparable to this group of phenomena."

Foerster seems to distinguish between the mercuric cyanide on the one hand and the fluoride ions on the other, calling the action of the first catalytic and the action of the second something else. There is a distinction between the two sets of substances; but it is not the one that Foerster makes. It is true that when one is using chlorine ion to raise the anode potential, there may be a change in this ion. Thus Müller[56] points out that when iodate is electrolyzed in presence of chloride, the process at the anode may consist chiefly in the formation of chlorine or hypochlorous acid and that these then react chemically to oxidize the iodate to periodate. While this may perhaps happen with chlorides, it certainly does not happen with fluorides. The essential part of the action is therefore catalytic, the fluoride remaining unchanged at the end of the run. As I see it, the two substances, mercuric cyanide and

[49] Elbs and Schönherr: Zeit. Elektrochemie, **2**, 250 (1895).
[50] Müller and Scheller: Zeit. anorg. Chem., **48**, 112 (1905).
[51] Müller: Zeit. Elektrochemie, **7**, 516 (1901); **10**, 64 (1904).
[52] Hofer and Moest: Liebig's Ann., **323**, 284 (1902).
[53] Skirrow: Zeit. anorg. Chem., **33**, 25 (1903).
[54] Müller: Zeit. Elektrochemie, **10**, 753 (1904).
[55] Müller: *Ibid*, **10**, 776 (1904).
[56] Zeit. Elektrochemie, **10**, 753 (1904).

potassium fluoride, both act as poisons but they poison different reactions. The mercuric cyanide cuts down the adsorption of thiosulphate and therefore decreases the oxidation and increases the polarization. To get the same current one must increase the voltage. There is no evidence in Thatcher's experiments that mercuric cyanide increases the over-voltage at the anode and Foerster assumes that it does not. In the case of potassium fluoride we do have an increase in the over-voltage, which means therefore an increase in the monatomic oxygen. If potassium fluoride poisons the anode for the reaction $2O \rightarrow O_2$, it would produce just the effects which we actually get and this is therefore a satisfactory working hypothesis for the time being. Whether it actually does retard this reaction is a matter to be tested experimentally. We ought to find a distinct relation between the substances which poison the hydrogen peroxide catalysis and those which change the over-voltage of a platinum anode.

Foerster says that we have nothing equivalent to this at the cathode; but I think that that is merely because we have not looked for it. There is only a very slight over-voltage with a smooth platinum cathode in sulphuric acid solution. There is a very large over-voltage with smooth platinum in a cadmium sulphate solution. It seems to me probable that one could get any intermediate value by taking a suitable mixture of cadmium sulphate and sulphuric acid or zinc sulphate and sulphuric acid. Nobody has ever tried this consciously because there was no especial reason for doing it, though the addition of a copper salt in the electrolytic determination of nitric acid is very possibly a case in point where the thing was done empirically.

The general conclusions to be drawn from this paper are:

1. Since contact catalysis involves adsorption, a decrease in the adsorption will mean a decrease in the reaction velocity, other things remaining the same.

2. The adsorption may be decreased by the presence on the solid catalytic agent of a film of solid, liquid, or gas.

3. Very small amounts of grease will keep palladium from taking up hydrogen.

4. Carbon monoxide cuts down the catalytic action of platinum on oxyhydrogen gas and is itself oxidized slowly.

5. The preferential oxidation of carbon monoxide is not the reason for its poisoning the catalytic action of platinum on oxy-hydrogen gas.

6. Carbon monoxide is retained tenaciously by platinum, and carbon monoxide practically destroys the catalytic action of platinum on hydrogen and ethylene.

7. Carbon monoxide undoubtedly decreases the adsorption of hydrogen and ethylene by platinum, though there are no direct measurements to show this.

8. Since the presence of the reaction products will decrease the rate at which the reacting substances will diffuse to the catalytic agent and since the reaction products may decrease the adsorption of the reacting substances, the reaction products may decrease the reaction velocity if they are not removed.

9. The reaction between carbon monoxide and oxygen in presence of platinum is accelerated by the addition of caustic potash to remove the carbon dioxide.

10. Sulphur trioxide decreases the rate at which sulphur dioxide and oxygen react.

11. If one reacting substance decreases the adsorption of the other very much, presence of an excess of the first may decrease the reaction velocity.

12. When a mixture of carbon monoxide and oxygen is catalyzed by quartz glass, an increase in the concentration of the oxygen increases the reaction velocity, but an increase in the concentration of the carbon monoxide decreases it.

13. Since the poisoning of the hydrogen peroxide catalysis is due to selective adsorption, there is no necessary reason why the same substances should poison both platinum and haemase or should poison them to the same extent. On the other hand there is no reason why there should not be a certain parallelism, and that is all that actually occurs.

14. When a colloidal metal is kept in suspension by the addition of gelatine or other substance, the stabilizer may cut down the adsorption and consequently may tend to decrease the reaction velocity.

15. If the retarding effect due to the stabilizer is greater than the accelerating effect due to increased surface, a stabilized col-

loidal metal may be less effective than a pulverulent metal which is not handicapped by a stabilizer.

16. The action of mercuric cyanide, etc., in cutting down the electrolytic oxidation of thiosulphate is probably due in part to its decreasing the adsorption of thiosulphate at the anode.

17. The action of fluoride ions, etc., in increasing the anode over-voltage is undoubtedly due to the fact that they decrease the catalytic action of the anode on the reaction $2O \rightarrow O_2$

18. An increase in over-voltage at the cathode could undoubtedly be obtained by taking a suitably acidified solution of zinc sulphate or cadmium sulphate. It is possible that the addition of copper salts in the electrolytic determination of nitrates is analogous to the addition of chloride or fluorides in the electrolytic formation of persulphates.

Cornell University.

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FRACTIONAL COMBUSTION

By Wilder D. Bancroft.*

[Abstract.]

The author discusses the general question of which of two combustible gases, mixed together, will burn the more rapidly, and the influence of a catalytic agent on the course of this phenomenon. For instance, hydrogen burns more readily than methane or ethylene in the presence of platinum. The influence of copper oxide, chamotte, and boro-silicate glass are reviewed. Hydrogen and carbon monoxide mixtures are similarly discussed. The specific effect of a catalytic agent in influencing these preferential combustions probably disappears at very high temperatures, where the reaction velocities become very high.

We may consider platinum black saturated with hydrogen, carbon monoxide, ethylene, methane, or oxygen as a hydrogen, carbon monoxide, ethylene, methane, or oxygen electrode and from a measurement of electromotive forces, we can determine which one of the first four gases should oxidize the most readily in presence of platinum. So long as we assume that no one of the gases interferes with any of the others in a mixture we can predict what a mixture will do. If, however, a mixture does not behave in the way it should, there is a factor which we have overlooked in our calculation of the electromotive forces. If we consider the platinum, as we usually do, as a means of letting the chemical affinities have free play, the relative oxidizability of the different gases should be the same in presence of and in absence of platinum. If there is a difference in the two cases, the question at once arises to what extent the electromotive force calculated from the free energy of hydrogen and oxygen is really the true electromotive

* Professor of Chemistry, Cornell University.

force of the hydrogen and oxygen gas cell. It is not my intention to answer either of these questions in this paper. I merely bring them up to show that a study of the theory of fractional combustion may be essential to a proper understanding of some electrochemical problems.

Many years ago Henry* made an interesting study of the effect of platinum on the fractional combustion of gases. Hydrogen and oxygen combine readily at ordinary temperatures in presence of platinum; carbon monoxide and oxygen react extremely slowly under the same circumstances; and neither ethylene and oxygen nor methane and oxygen react at all. When a mixture of methane and oxyhydrogen gas is exposed to platinum, the hydrogen burns and the methane does not. When a mixture of ethylene and oxyhydrogen gas is placed in contact with platinum some of the ethylene always burns. When a mixture of carbon monoxide with oxyhydrogen gas is exposed to platinum, there is a very slow reaction, the carbon monoxide burning more readily than the hydrogen.

Hydrogen is adsorbed by platinum more strongly than ethylene and consequently it tends to burn first. Since ethylene does not burn at all under the same circumstances, it is possible, though not probable, that the combustion in presence of hydrogen is due to the oxygen passing through some intermediate stage in which it oxidizes the ethylene. That would make this an induced reaction. It seems to me more probable that we are dealing with a case of local heating, the ethylene burning at the higher temperature thus produced. This is the more probable since more ethylene is burned the greater the ratio of oxyhydrogen gas and the greater the ratio of oxygen. In fact Henry found that ethylene, when mixed with sufficient oxygen, began to burn in contact with platinum sponge at about 250° and was converted completely into carbon dioxide at about 270°. Lunge and Harbeck[1] have shown that carbon monoxide is retained very tenaciously by platinum. It should therefore burn before hydrogen, and so it does. Methane is undoubtedly adsorbed much less than either hydrogen or carbon monoxide, and this is in line with the fact that it is burned less readily under these circumstances.

* Phil. Mag., **65**, 269 (1825).

[1] Zeit. anorg. Chem., **16**, 50 (1898).

Henry based a method of gas analysis on these observations. He found that by regulating the temperature carefully "the action of oxygen upon several gases (carbonic oxide, olefiant gas, and carburetted hydrogen, for example) may be made to take place in succession; and by removing the carbonic acid formed at each operation, it may be ascertained how much of each of the first two gases has been decomposed. The carburetted hydrogen indeed always remains unchanged, and its quantity must be determined by firing it with oxygen by the electric spark. If hydrogen also be present, it is difficult to prevent the olefiant gas from being partially acted upon; but this is of little consequence, as I had shown that it is easy to remove that gas in the first instance by chlorine."

This method of Henry's was afterwards worked out in more detail by Hempel,[2] substituting palladium for platinum. A mixture of hydrogen with an excess of oxygen burns completely when passed over palladium. The reaction begins spontaneously at ordinary temperatures, but so much heat is evolved that the palladium glows and the gases explode in case they are present in the ratio to form water. When methane and oxygen are passed over palladium at 100° no combination takes place. The reaction starts at about 200°. Thus there was a contraction of 3 c.c. when a mixture of 29.3 c.c. methane and 70.6 c.c. oxygen was passed several times over palladium at about 200° to 220°. When a mixture of hydrogen, methane, and air is passed over palladium at ordinary temperatures or at 100°, the hydrogen alone is burned and the methane remains unchanged, provided precautions are taken so that the palladium does not heat up too much.[3]

If a mixture of hydrogen and methane is passed over copper oxide at 250°, the hydrogen is burned completely, while the methane is not oxidized at all.[4] This method is said[5] to be distinctly superior to the fractional combustion of hydrogen by palladium or palladium asbestos because no air or oxygen is added to the combustible gas, which makes it possible to pass the total gas residue through the tube with consequent gain in the accuracy of the analytical results.

[2] Ber. deutsch. chem. Ges., **12**, 1006 (1879).
[3] *Cf.* Hempel: Zeit. angew. Chem., **25**, 1841 (1912).
[4] Jäger: Jour. Gasbeleuchtung, **41**, 764 (1898).
[5] Dennis: Gas Analysis, 199 (1913).

In view of the comparative readiness with which methane can be separated from hydrogen by the fractional combustion of the latter gas, it is not surprising that people came to the conclusion that hydrogen always burns more readily than methane. From experiments on the burning of illuminating gas, Landolt[6] concluded that in the flame "the gases disappear in the order of their combustibility. Hydrogen burns more readily than any other gas and consequently disappears the soonest in the flame. Marsh gas disappears somewhat more slowly, and the denser hydrocarbons disappear latest, burning chiefly in the upper half of the flame." It has been pointed out by Bone[7] that Landolt's results are vitiated by the fact that he used a platinum tube of narrow bore, fixed along the vertical axis of the flame for the withdrawal of the partly burned products.

TABLE I.

Volumes Before Firing			Ratio of $H_2O:CO_2$
CO	H_2	O_2	
7.36	2	1	0.85
5.98	2	1	1.08
4.87	2	1	1.28
5.03	2	1	1.29
3.90	2	1	1.53
2.82	2	1	1.83

Henry's experiments showed that carbon monoxide burned before hydrogen when in contact with platinum at relatively low temperatures and that hydrogen burned before carbon monoxide when the gases were exploded by an electric spark. This last remark is confirmed by Bunsen's experiments[8] given in Table I. More hydrogen is burned than carbon monoxide until the ratio of the second to the first is at least six. Henry[9] is quite explicit that the preferential burning of carbon monoxide in presence of platinum at 150° to 170° and the preferential burning of hydrogen when the gases are fired by the electric spark is merely a question

[6] Pogg. Ann., **99**, 411 (1856).
[7] Phil. Trans., **215**, 297 (1915).
[8] Bunsen: Ges. Abh., **2**, 586 (1904).
[9] Phil. Mag., **65**, 277 (1825).

of temperature. Presumably Bunsen and Landolt held the same view because they do not comment on the low temperature results, which they probably would have done had they considered these results abnormal.

A new light has been thrown upon the phenomena by Bone[10] and his co-workers. Bone was primarily interested in the way in which a hydro-carbon burns in a supply of oxygen insufficient to oxidize it completely to carbon monoxide and steam; but, as is usually the case, the scope of the investigations soon became much broader. A mixture of oxygen and the gas to be studied was sealed in cylindrical bulbs of borosilicate glass with capillary ends; the bulbs were then kept at constant temperatures between 300° and 400° for several days in an air bath until the whole or a part of the oxygen had disappeared. The lowest temperature at which a reaction between methane and oxygen could be observed after the lapse of two or three weeks was 300°. At 325° the rate of reaction was much greater and at 350° in some experiments the whole of the oxygen disappeared within three or four days, while at 400° the oxidation was always completed in a single day. Hydrogen and oxygen at 350° do not combine to any appreciable extent within a week or two, provided the glass surface with which the gases are in contact remains perfectly smooth. At 400° the formation of water may be recognized within a week, but hardly within three days, whereas at this temperature the whole of the oxygen reacts with methane within a single day. Methane therefore combines with oxygen between 300° and 400° with an enormously greater velocity than does hydrogen under the same conditions. In a later paper Bone[11] says that "under similar conditions ethane burns much more rapidly than methane, and in borosilicate glass bulbs both hydrocarbons are oxidized at temperatures much below the limiting temperature (about 400°) at which, under similar conditions, steam is formed appreciably from electrolytic gas." In a lecture before the Royal Institution, Bone[12] showed that when mixtures corresponding to $C_2H_2 + 2H_2 + O_2$ and $C_2H_4 + H_2 + O_2$ are exploded there is no deposition of carbon and only a very slight formation of steam. The hydrocarbon has been burned to carbon monoxide and hydrogen, leaving

[10] Jour. Chem. Soc., **81**, 535 (1902) *et seq.*
[11] Jour. Chem. Soc., **85**, 694 (1904).
[12] Proc. Roy. Inst., **19**, 82 (1908).

the hydrogen of the original mixture absolutely untouched by the oxygen.

Experiments on explosions in bombs[13] showed that even at high temperatures there is preferential burning of methane. Bone concludes from experiments with the mixture $CH_4 + 2H_2 + O_2$ that "the ratio of the affinities [really reaction velocities] CH_4 : H_2 is of the order 20 or 30 to 1 at least, and probably higher if the influence of the walls of the containing vessel could be eliminated."

From the experiments of Henry, Landolt, and Bone, we see that hydrogen burns before methane at low temperatures in contact with platinum and at high temperatures when the gases are drawn off through a platinum tube. We see also that methane burns before hydrogen at moderate temperatures in borosilicate glass bulbs and also when the gases are fired by an electric spark. It is quite clear that a surface phenomenon is an important factor in changing the results. In Henry's experiments the platinum is unquestionably the cause of the preferential burning of the hydrogen and it seems probable that this is also true for Landolt's experiments. The existence of the water gas equilibrium with hydrogen as one of the constituents is fairly strong evidence that hydrogen is more stable than methane. Bone's results may therefore be considered as approaching at high temperatures the results due to free burning away from a surface. This is the more probable because he always finds a greater relative oxidation of methane in the larger bomb where the ratio of surface to volume is less. The existence of a surface factor is also shown by Bone's observation that about eighty percent combustion of hydrogen occurs when the mixture $CH_4 + O_2 + 2H_2$ is passed over chamotte[14] at 500°. I do not know enough about the adsorbing properties of chamotte to know just why it acts as it does; but this is a very striking reversal.

Since we can get almost complete preferential burning either of hydrogen or methane at moderate temperatures, depending on the nature of the surface, it is probable that a more extended study of different surfaces would give us all the intermediate values

[13] Bone: Phil. Trans., **215** A, 298 (1915).
[14] Bone: Ber. deutsch. chem. Ges., **46**, 12 (1913).

for the distribution of oxygen between methane and hydrogen just as Sabatier and Mailhe[15] have found all sorts of gradations between the reactions $CH_3CH_2OH = C_2H_4 + H_2O$ and $CH_3CH_2OH = CH_3CHO$ and H_2 when alcohol is passed over different heated metals or oxides. While I have not been able to find any definite studies along this line, there are some indications that special results might be obtained with special solids.

Calvert[16] showed that oxygen adsorbed by boxwood charcoal oxidizes ethylene to carbon dioxide and water at ordinary temperatures. This is not surprising because charcoal adsorbs ethylene strongly. Since charcoal has little or no effect in causing hydrogen and oxygen to combine, it is probable that we should get a preferential burning of ethylene in case a mixture of ethylene, hydrogen, and oxygen were brought in contact with charcoal. While this experiment seems not to have been tried, we do know that charcoal does seem to promote oxidation of carbon compounds.

In the form of wood ashes, charcoal is often used in outhouses and elsewhere to eliminate unpleasant odors. The charcoal first adsorbs the odoriferous substances and then accelerates their oxidation, by oxygen which is adsorbed from the air. This dual function of the charcoal was pointed out clearly by Stenhouse[17] in 1854.

It is interesting to note that Bone's experiments on surface combustion led him to the conclusion that adsorption played an important part in the phenomenon. Bone[18] began by a study of the combination of hydrogen and oxygen in contact with hot surfaces of porcelain, magnesia, silver, gold, platinum, nickel, calcined spathic iron ore (containing 79.0 percent ferric oxide and 14.5 percent manganous oxide), nickelous oxide, and copper oxide. With each of these surfaces the rate is proportional to the pressure of the dry gas when electrolytic gas is used, is approximately proportional to the partial pressure of hydrogen when either gas is present in excess, and is increased by a previous treatment with hydrogen. In the case of porcelain, Bone decides that "porous porcelain occludes or condenses both hydrogen and oxygen at rates which depend to some extent upon the physical condition and past

[15] Ann. Chim. Phys. (8), **20**, 341 (1910).
[16] Jour. Chem. Soc., **20**, 293 (1867).
[17] Pharm. Jour., **13**, 454 (1854).
[18] Phil. Trans., **206** A, 1 (1906).

history of the surface. In general, however, whereas in the case of oxygen the process is extremely rapid and the surface layer is soon saturated, the occlusion of hydrogen is slower and the limit of saturation much higher. Combination between the occluded gases occurs at a rate either comparable with, or somewhat faster than, the rate at which the film of occluded oxygen is renewed, but considerably faster than the rate of occlusion of hydrogen."

The behavior of magnesium, "together with the fact that the material occludes hydrogen but not oxygen at red heat, shows that the catalytic action must be referred to the power of occluding hydrogen." The catalytic action of the four metals examined is due primarily "to an association of the surface with hydrogen—in the case of silver probably to the formation of an unstable hydride, in the other three cases to an occlusion of the gas. * * * The evidence in favor of the 'occlusion' theory is particularly strong in the case of nickel, where the 'normal' condition of activity can be reduced almost to the vanishing point by prolonged exhaustion at a comparatively low temperature. Moreover, the subsequent recovery of activity can be demonstrated by circulating successive charges of electrolytic gas over the surface under conditions which entirely preclude any chemical action of either of the reacting gases on the metal." "Considering the results obtained with the three different reducible oxides as a whole, they seem to be quite incompatible with any purely chemical explanation of the catalytic process. In the case of copper oxide it would appear to involve the condensation of a film of 'active' oxygen on the surface, and that this film actually protects the catalyzing oxide from the attacks of the hydrogen, which would otherwise reduce it energetically. At low pressures the film becomes too attenuated to ensure complete protection, and in consequence the formation of steam is accelerated by reason of hydrogen penetrating through onto the oxide and reducing it. In conformity with this idea, the rate of steam formation when the gases are not present in combining ratios was found to be proportional to the partial pressure of the oxygen.

"In the cases of the other reducible oxides examined, the fact that the gases combined at moderate temperatures, without permanently altering the surface, and at rates always far in excess

of the rates at which either the surface was reduced by hydrogen, or the reduced surface re-oxidized, does not harmonize with the purely chemical theory. It is difficult to resist the conclusion that in these cases also the catalytic action is due primarily to a physical condensation of one or other of the reacting gases at the surface. Since the rate of combination was always proportional to the partial pressure of the hydrogen in all experiments where the gases were not present in combining ratios, it would appear to be the hydrogen which is condensed and so rendered active."

The fact that the catalytic combustion depends on an intimate contact of the reacting gases with the surface is shown by the appearance of a piece of silver gauze before and after it had acted as catalytic agent for hydrogen and oxygen at 400°. The wires were originally very smooth, but after using they became quite rough. Bone[19] points out that at very high temperatures the specific nature of the solid seems to be relatively unimportant. At such temperature one would get practically no displacement of equilibrium by the catalytic agent because the reaction in the gas phase takes place so rapidly that the final equilibrium is that of the gas phase and not that of the catalytic agent.

The general conclusions to be drawn from this paper are:

1. At low temperatures the nature of the catalytic agent may determine which of two combustible gases will burn the more rapidly.

2. As might be expected, hydrogen burns more readily than methane or ethylene in presence of platinum.

3. From Landolt's experiments the same seems to be true in the flame provided a platinum tube is inserted; but these experiments should be repeated.

4. In presence of copper oxide at 250° all the hydrogen in a mixture of hydrogen and methane can be burned without any of the methane being decomposed.

5. In presence of chamotte at 500° hydrogen burns much more readily than methane. So little is known about the adsorption by chamotte that this result could not have been predicted.

6. In borosilicate glass bulbs at 300° to 400° methane, ethane, ethylene, and acetylene are oxidized much more rapidly than hydrogen or carbon monoxide.

[19] Ber. deutsch. chem. Ges., 46, 14 (1913).

7. When mixtures of methane, oxygen, and hydrogen or carbon monoxide are exploded by an electric spark the methane burns much more rapidly than either the hydrogen or the carbon monoxide.

8. Depending on conditions, either hydrogen or carbon monoxide will burn the more readily.

9. Since charcoal causes ethylene to be oxidized to carbon dioxide and water, and since charcoal has very little effect on a mixture of hydrogen and oxygen, it is probable that charcoal would cause preferential burning of ethylene in a mixture of ethylene and hydrogen.

10. By suitable selection of the catalytic agent it should be possible to get all intermediate stages from complete burning of methane to complete burning of hydrogen.

11. Since relatively more methane was burned in the explosion experiments the larger the bomb, it seems probable that methane burns more readily than hydrogen at high temperatures and in the absence of solid catalytic agents.

12. At very high temperatures the specific effect of the solid catalytic agent must become negligible because the reaction will take place so rapidly in the gas phase that the equilibrium reached will be that of the gas phase and not that of the catalytic agent.

Cornell University.

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THEORY OF CONTACT CATALYSIS.

By Wilder D. Bancroft.*

[*Abstract.*]

The facts and the various theories of catalytic action are reviewed and discussed from the standpoint of adsorption upon the catalyst as the determining factor controlling catalytic action.

This viewpoint is applied to the discussion of the action of a catalyst upon mixtures. This is extended to electrolytic reactions in which combinations of the solvent with the ions are considered. The final conclusion is that a solid catalytic agent is equivalent to a solvent, and therefore may displace the equilibrium of the system.

A change in the nature of the cathode metal will often have a marked effect on the nature of the products of electrolysis. Typical instances are the reduction of nitrobenzene to aniline with a copper cathode and of nitric acid to hydroxylamine with a mercury cathode. Since these differences are due to contact catalysis, a discussion of the general theory may be of interest, even though most of the applications are not electrochemical in nature.

For a solid to act catalytically it must adsorb one or more of the reacting substances, thereby increasing the concentration at the surface. This increased concentration will necessarily cause an increase in the reaction velocity quite apart from any special catalytic action which the solid may exert. There may be catalytic action in all cases; but there must be in some, so it will be better to consider a few cases in which the results may perhaps be due solely to increase of concentration, and we can then take up the more interesting cases in which catalysis must occur.

* Professor of Chemistry, Cornell University.

Döbereiner showed that when hydrogen is allowed to pass over spongy platinum in presence of air, the hydrogen takes fire. So far as I know, nobody has ever compressed oxyhydrogen gas so that it occupied the volume of liquid water. If no combination took place under these circumstances, it would be necessary to ascribe a special catalytic action to the platinum. Until that experiment is tried, it is possible that the adsorbed hydrogen reacts with the adsorbed oxygen or with platinum hydroxide by virtue of the increase in concentration. In the case of SO_2 and O_2, Hempel[1] obtained a yield of 65 percent SO_3 without any catalytic agent by working at a pressure of 30-40 atmospheres. On the other hand the fact that charcoal has practically no effect in causing hydrogen and oxygen to combine indicates that some other factor is important in addition to increased surface concentration. The enormous effect of change of concentration is shown clearly in the experiments of Berthelot and Péan de St. Gilles on the ester formation. At the same temperature the reaction between liquid alcohol and liquid acid takes place about one thousand times as fast as the reaction between gaseous alcohol and gaseous acid.[2]

The more interesting cases of contact catalysis are those in which we know that some other factor enters besides that of increased concentration. If a given substance decomposes in one way in presence of one catalytic agent and in another way in the presence of another catalytic agent, the difference in the reaction products cannot be explained on the hypothesis of increased surface concentration. Since we have selective adsorption from a gaseous mixture, we may also have selective adsorption of the reaction products, and this may cause one reaction to predominate at the expense of another. It is usually assumed that a catalytic agent cannot displace the equilibrium; but when people take this ground they are almost invariably considering cases where the reaction takes place with measurable velocity in absence of the catalytic agent, though they are not right under these conditions because they do not consider the possibility of a solvent acting as a catalytic agent.

[1] Ber. deutsch. chem. Ges., **23**, 1455 (1890).
[2] Ostwald: Zeit. Elektrochemie, **7**, 1002 (1901).

Whether a solvent can be considered as a catalytic agent or not depends on the definition of a catalytic agent. Ostwald[3] says that "when a substance by its mere presence alters the velocity of a reaction, and may be recovered unaltered in nature or amount at the end, it is called a catalytic agent or catalyzer, and the phenomenon is called catalysis." Nernst's definition[4] is practically identical, being based on Ostwald's. Catalysis "means an increase in velocity of reaction caused by the presence of substances which do not take part in it (or only to a secondary extent), although the reaction is capable of taking place without their presence." Since a solvent changes the reaction velocity without necessarily taking part in the reaction, a solvent may be considered as a catalytic agent. This is recognized explicitly by Ostwald.[5]

"Catalysis, as just defined, is an uncommonly widespread phenomenon, which actually occurs in all cases in which the rate of a chemical reaction can be measured. A splendid example of this is to be found in the well-known experiments of Menschutkin. For a number of different reactions he has proved that the reaction velocity may vary within wide limits depending on the solvent used. We must therefore call these actions of the solvents catalytic actions. This characterization does not, of course, exclude the possibility that it may be shown that compounds are formed between the solvents and the reagents so that the changes in reaction velocity can be referred to changes in the active masses. All intermediate stages can be found between cases like these and cases in which a very small amount of an added substance changes the reaction velocity very much. This latter class of cases is the only one that hitherto has been considered as catalysis; but the difference is merely a quantitative one, and it is improper, when studying these things systematically, to exclude those cases in which the acceleration is relatively small."

Bigelow[6] says practically the same thing in somewhat different words. "Instances of catalysis may be multiplied almost without number. A given reaction occurs at different rates in different solvents. The solvent remains unaltered at the end of the reac-

[3] Zeit. Elektrochemie, 7, 998 (1901); Lehrbuch allgem. Chem., 2, II, 248 (1902). Quoted from Bigelow: Theoretical and Physical Chemistry, 365 (1912).
[4] Nernst: Theoretical Chemistry, 590, 616 (1916).
[5] Zeit. Elektrochemie, 7, 998 (1901).
[6] Theoretical and Physical Chemistry, 366 (1912).

tion and hence all the effects must be reckoned with catalytic actions. Menschutkin[7] measured the velocity of the reaction,

$$N(C_2H_5)_3 + C_2H_5I = N(C_2H_5)_4I$$

in a variety of solvents. In hexane the velocity constant, *k*, had the value 0.000180; in heptane, 0.000235; in benzene, 0.00584; in ethyl alcohol, 0.0433; in benzyl alcohol, 0.133; and intermediate values in other solvents. The velocity is nearly one thousand times as great in benzyl alcohol as in hexane.

"The difference in velocity of the above reaction in hexane and heptane is but slight, though the quantity of the catalytic agent is large, it being the solvent. The most fascinating instances of catalysis are those in which small traces of the catalyzer produce great effects, and we are rather too apt to assume that these alone are instances of catalysis. Some of the quantitative measurements are indeed striking. For instance, Bredig,[8] studying the increased velocity with which hydrogen peroxide decomposes in the presence of colloidal platinum, was able to detect such increase when the concentration of the platinum was but one seventy-millionth of a symbol weight per liter. Again Titoff[9] found that the velocity with which sodium sulphite was oxidized was increased 30 percent by the presence of one-thousandth of a formula weight in grams of copper sulphate per liter."

The effect of the solvent on the rate of decomposition of xanthogenic acid into carbon bisulphide and alcohol has been studied by von Halban and Kirsch.[10] The reaction may be written

$$HSSCOC_2H_5 = CS_2 + C_2H_5OH.$$

If the reaction velocity constant for carbon bisulphide as solvent be taken as unity, we get the following values at 25°: Carbon bisulphide, 1.00; ligroin (hexane), 1.50; chloroform, 3.64; benzene, 5.15; nitrobenzene, 315; ether, 485; acetone, 25,980; alcohol, 1,000,000. In other words, the decomposition takes place a million times as rapidly in alcohol as in carbon bisulphide, the two decomposition products.

[7] Zeit. phys. Chem., 6, 41 (1890).
[8] *Ibid.*, 31, 258 (1899).
[9] *Ibid.*, 45, 641 (1903).
[10] *Ibid.*, 82, 325 (1913).

It is well known that a solvent may displace equilibrium. Cundall[11] has made a quantitative colorimetric study of solutions of N_2O_4 in different solvents at concentrations of nearly 3 grams N_2O_4 per 100 cc. Ostwald's calculations[12] of these data are given in Table I. The percentage dissociation varies for the extreme cases in the ratio of about one to six. The dissociation in the vapor would be much greater for the same concentrations than in any of the liquids, for Ostwald calculates that the dissociation

TABLE I.

Dissociation of N_2O_4 in Solution.

Solvent	Liters per Mol.	Percentage Dissociation		
		10°	20°	30°
Chloroform	3.36	0.36	0.68	1.24
Methyl chloride	3.39	0.29	0.56	1.06
Carbon tetrachloride	3.48	0.46	0.82	1.43
Ethylene chloride	3.45	0.20	0.41	0.70
Ethylidene chloride	3.13	0.32	0.60	1.09
Benzene	3.33	0.22	0.43	0.80
Chlorobenzene	3.40	0.30	0.56	0.99
Brombenzene	3.22	0.31	0.55	0.94
Bromoform	3.39	0.23	0.52	0.98
Ethyl bromide	2.93	0.33	0.63	1.14
Ethylene bromide	3.34	0.37	0.59	0.95
Silicon tetrachloride	3.34	0.71	1.22	2.43
Carbon bisulphide	3.20	0.57	1.05	1.79
Acetic acid	3.25	0.11	0.22	0.40

constant for the gas is about one hundred times that for nitrogen peroxide in chloroform at 20° and about four hundred times that for nitrogen peroxide in chloroform at 10°. No actual measurements have been made on nitrogen peroxide vapor at the corresponding pressures of 6-7 atmospheres.

An even more striking case is that of hydrochloric acid and water, where there are practically no ions in the vapor phase and the hydrochloric acid is highly dissociated in the liquid phase. As Nernst[13] points out, "the assumption that water has an exceptional power of causing dissolved substances to dissociate elec-

[11] Jour. Chem. Soc., **59**, 1076 (1899); **67**, 794 (1895).
[12] Ostwald: Lehrbuch allgem. Chemie, **2**, II, 602 (1892).
[13] Zeit. phys. Chem., **8**, 139 (1891).

trolytically can be replaced by another that the solubility coefficient of ions in water is exceptionally large. Possibly this latter assumption may seem less abhorrent to some than the first one; but the one is merely another way of wording the other." The general effect of the solvent in displacing equilibrium is to cause a greater relative formation of the system which is the more soluble in the solvent.

In the case of nitrogen peroxide and a solvent, we get one equilibrium in the vapor and another equilibrium in the solution; but if we had a hypothetical case in which the reaction took place immeasurably slowly in the vapor phase and relatively rapidly in the solution, the equilibrium in the vapor would be reached only by volatilization from the solution, and this might give rise to some rather interesting results. Something similar to this has been suggested by Bredig and discussed by Ostwald.[14] "Let us imagine that a portion of a gaseous system is converted at the same temperature into the liquid state or is given a density comparable with that of the liquid state. The reaction will take place rapidly in this portion and the liquefied reacting substances will change into the corresponding reaction products. If the cause for the liquefaction or for the increase in density is such that more of the reacting substances are liquefied or given an increased density as fast as the preceding portions have reacted, these would react rapidly and the process would be repeated, with the result that we have an accelerated reaction. Such an action of platinum on gases is quite conceivable. I do not mean by this to say that this is the way that platinum catalyses actually occur, but merely to point out a possible way in which they might occur. This would then be the simplest case of the accelerating intermediate reactions to which I have previously referred.

"Professor Bredig has recently pointed out to me that the mechanism of such an acceleration becomes intelligible if we consider a liquid medium in which is suspended a small drop of a second liquid. If this suspended liquid has the property of making the substances dissolved in it react more rapidly than in the main mass, the bulk of the reaction will take place there. The reaction products will diffuse into the outer liquid and new portions of the reacting substances will diffuse into the drop. In

[14] Zeit. Elektrochemie, 7, 1002 (1901).

this way practically all of the reacting substances will diffuse into the suspended liquid and will react there, the result being an acceleration of the reaction.

"According to Bredig, what has been outlined here may perhaps be applicable to the case where the catalytic agent is present in the colloidal state in the liquid. It is known that Professor Bredig and his pupils in a series of splendid investigations have shown and measured the manifold and energetic catalytic actions due to colloidal platinum and other colloidal metals prepared by the Bredig process. He has also emphasized repeatedly that the enzymes, those natural and very effective catalytic agents, are also always in a state of colloidal solution or suspension.

"These considerations have no claim to being anything more than suggestions which can be tested experimentally. I cannot refrain from pointing out that it has only been possible to make suggestions which can be tested scientifically, since we adopted the conception of a catalytic agent being an accelerator. Let one just try to do anything like this on the assumption of molecular vibrations."

The same matter has been discussed by Höber.[15] "When ethyl acetate is dissolved in benzene, it does not change perceptibly with time.[16] If one adds an aqueous acid solution as a ferment, the catalytic decomposition takes place. Bredig,[17] who first developed the point of view, devised the following artificial enzyme action. When tri-ethylamine is added to a solution of methyl acetate in benzene at 25°, the base does not saponify the ester under these conditions. If one forms an emulsion by shaking the mixture with 2.5 percent water, the base and the ester dissolve freely in the water as the better solvent and the ester is saponified rapidly by the base, which is highly dissociated in water. The drops of water therefore act as an enzyme, increasing the reaction velocity without themselves taking part appreciably in the reaction. In this way we gain a conception of the way in which catalytic agents act, which is quite different from the ordinary point of view. The enzyme does not combine with the substrate to form an intermediate product in an intermediate reaction, but owes its catalytic action to its being a better reaction medium."

[15] Physikalische Chemie der Zelle und der Gewebe, 4th Ed., 703 (1914).
[16] [It is not clear from this statement what reaction Höber has in mind.]
[17] Anorganische Fermente, 92 (1901).

Neither Ostwald nor Bredig has drawn the important corollary that the equilibrium will be determined primarily by the solvent in which the reaction takes place and that it is therefore theoretically possible to obtain one set of reaction products with one liquid as catalytic agent and another set of reaction products with another liquid as catalytic agent.

If we consider a solid catalytic agent as equivalent to a drop of liquid, as suggested by Bredig, and the formation of an adsorption film as equivalent for our purposes to solution, it becomes possible theoretically to get different reaction products with different solid catalytic agents, just as it is possible theoretically to get different reaction products with different solvents. If we have a reaction which takes place extremely slowly in the vapor phase and relatively rapidly in or at the surface of a solid catalytic agent, we shall get the equilibrium in or at the surface which corresponds to that solid catalytic agent hypothetically as equivalent to a solvent. This equilibrium will not necessarily be the same as the theoretical equilibrium in the vapor phase any more than the dissociation of nitrogen peroxide in chloroform solution is the same as the dissociation of nitrogen peroxide in the vapor phase. When the reaction products diffuse from the surface of the solid catalytic agent into the vapor phase, they do not react in measurable time by definition, and consequently we get in the vapor phase an apparent equilibrium corresponding to that at the surface of the solid catalytic agent, and therefore varying perhaps with different solid catalytic agents.[18]

The conception of a solid catalytic agent as being equivalent to a drop or film of liquid is due originally to Bredig[19] and has also been made use of by Bodenstein,[20] who considers the reacting substances as actually dissolved in the platinum instead of adsorbed at the surface. In so far as the platinum is actually a solvent and not merely equivalent to a solvent, the possibility of an apparent displacement of equilibrium is beyond question and the alleged violation of the second law of thermo-dynamics becomes an imaginary one.

We have now to consider what the direction of the reaction will be when several reactions are possible. We have already

[18] *Cf.* Bancroft: Jour. Phys. Chem., **12**, 138 (1908).
[19] Zeit. Elektrochemie, **7**, 1002 (1901).
[20] Zeit. phys. Chem., **46**, 736 (1903).

seen that the solvent displaces the equilibrium so that the ratio of the more soluble system to the less soluble system in the solution is increased. This can be put in a more general form which covers all cases by saying that there is a relative increase in the amount of the system the chemical potential of which is decreased relatively by the medium.[21] If one treats a substance with a dehydrating agent, the tendency of the substance to split off water is increased. A very interesting case coming under this general head is the decomposition of hydrazine in hot aqueous solution in presence of platinum.[22] Hydrazine may decompose in three different ways according to the reactions,

$$6N_2H_4 = 8NH_3 + 2N_2$$
$$6N_2H_4 = 6NH_3 + 3N_2 + 2H_2$$
$$6N_2H_4 = 4NH_3 + 4N_2 + 6H_2$$

The first reaction is the chief one when starting with hydrazine sulphate; the second one predominates when starting with free hydrazine; while the third one comes to the front in an alkaline hydrazine solution. There is more ammonia formed, the more acid the solution is. This illustration is given as being both typical and simple. Tanatar[23] found that a hot alkaline solution of hydroxylamine decomposed chiefly according to the equation $3NH_2OH = NH_3 + N_2 + 3H_2O$, while the same solution in presence of platinum decomposed according to the equation $4NH_2OH = 2NH_3 + N_2O + 3H_2O$. The change in the course of the reaction is due to the addition of the solid catalytic agent and not to any change in the alkalinity or acidity. Tanatar considers that part of the hydroxylamine oxidizes the platinum to an unspecified oxide, being itself reduced to ammonia, while the platinum oxide then oxidizes another portion of the hydroxylamine to nitrous oxide. This explanation seems absolutely impossible, and it will probably be wise not to offer any explanation for this case until more is known about the adsorption of nitrous oxide by platinum.

We are now in a position to predict that, when we are dealing with something more than a mere increase in concentration, we

[21] *Cf.* Miller: Jour. Phys. Chem., 1, 636 (1897).
[22] Tanatar: Zeit. phys. Chem., 40, 475; 41, 37 (1902).
[23] Zeit. phys. Chem., 40, 475 (1902).

may get different results with different catalytic agents. Under these circumstances the nature of the reaction will be determined by the specific adsorption of the reaction products by the catalytic agent in question.

When heated in presence of certain catalytic agents alcohol may be decomposed into aldehyde and hydrogen or into ethylene and water, to take two instances. We should expect catalytic agents which adsorb hydrogen strongly to cause the splitting to aldehyde and hydrogen, while catalytic agents which adsorb water readily should promote the decomposition into ethylene and water. As a matter of fact alcohol is decomposed nearly quantitatively into aldehyde and hydrogen when passed over heated nickel, which adsorbs hydrogen.[24] It is decomposed nearly quantitatively into ethylene and water when passed over heated alumina,[25] which must therefore adsorb water. Johnson[26] has shown that up to a certain point alumina adsorbs water so completely that phosphorus pentoxide takes no more out. When titanium oxide is used as the catalytic agent, both reactions take place simultaneously, and a yield of 84 percent ethylene and 16 percent hydrogen can be obtained under certain conditions. This would seem to imply that titanium oxide adsorbs both water and hydrogen. This was proved indirectly by Mr. Engelder in the Cornell laboratory. When aqueous alcohol is passed over titanium oxide, the water vapor cuts down the reaction which produces water and consequently the relative yield of acetaldehyde is increased over that obtained from absolute alcohol. When hydrogen is mixed with the alcohol vapor the relative amount of ethylene is increased. A somewhat similar result appears to have been obtained unconsciously by Berthelot[27] nearly fifty years ago. He heated formic acid at 260° without any specified catalytic agent and found that when only a third of the formic acid is decomposed the reaction appears to be $HCO_2H = CO + H_2O$. If all the formic acid is decomposed, the reaction is approximately $2HCO_2H = CO + H_2O + CO_2 + H_2$. This unexpected result can only be true in case the reaction $HCO_2H = CO_2 + H_2$ predominates during

[24] Sabatier and Senderens: Comptes rendus, **136**, 738, 921, 936, 983 (1903).

[25] Ipatieff: Ber. deutsch. chem. Ges., **34**, 3579 (1901); **35**, 1047, 1057 (1902); **36**, 1990, 2003, 2014, 2016 (1903).

[26] Jour. Am. Chem. Soc., **34**, 911 (1912).

[27] Ann. Chim. Phys. (4), **18**, 42 (1869).

the latter part of the decomposition, and this can happen only in case the original decomposition products check the initial reaction and thus permit the second reaction to come to the fore. It was this unexplained observation by Berthelot which gave me the clue to the present theory of contact catalysis.

When acetaldehyde and excess of hydrogen are passed over suitably heated nickel, the reaction is reversed and alcohol is formed. Similarly benzene and an excess of hydrogen forms cyclohexane in presence of nickel and nickel splits cyclohexane into benzene and hydrogen.[28] We can even go one step farther and can convert either benzene or cyclohexane into methane by treating with nickel and a sufficient excess of hydrogen. Zelinsky[29] has shown that palladium will split cyclohexane into benzene and hydrogen or will reverse the reaction under different conditions. Since the temperatures are different with palladium and with nickel, it is evident that we have a true displacement of equilibrium though no quantitative study has yet been made. Wilstätter and Hatt[30] have shown that when benzene is dissolved in cold acetic acid it can be converted into cyclohexane by the action of hydrogen in the presence of platinum black.

It is interesting to note that Jellinek[31] claims to have obtained more ammonia than corresponds to equilibrium conditions by heating nitrogen and hydrogen in presence of iron as catalytic agent. He points out that iron adsorbs ammonia very markedly. It is therefore theoretically possible that his data are correct, though they would have to be repeated before one would dare to lay much stress on them. Müller[32] electrolyzed a normal potassium nitrate solution using a porous diaphragm, a platinum anode 2 cm. x 5 cm. and a cathode of the same size, made of platinum, iron, or zinc. The cathode solution was stirred by a current of hydrogen. A current of one ampere was sent through the cell for three hours. In Table II are given the metal used as cathode, the voltage drop at the terminals, and the amount of ammonia formed.

[28] Sabatier and Senderens: Ann. Chim. Phys. (8) **4**, 336, 361 (1905).
[29] Ber. deutsch. chem. Ges., **44**, 3121 (1911).
[30] *Ibid.*, **45**, 1471 (1912); *Cf.* Willard: *Ibid.*, **45**, 2615 (1912).
[31] Zeit. anorg. Chem., **71**, 121 (1911).
[32] *Ibid.*, **26**, 43 (1901).

TABLE II.

Cathode Metal	Voltage Drop	Grams Ammonia
Smooth platinum	4.8	0.0416
Iron	4.4	0.1894
Zinc	4.5	0.1210

Müller himself comments on the fact that most ammonia was formed at the iron cathode, although the voltage drop was lowest in this case. It is probable, though not proved, that this is a catalytic action of the iron cathode and dependent on the adsorption of ammonia by iron.

The catalytic setting free of hydrogen has been studied by Sieverts[33] for the case of hypophosphorous acid. In aqueous solutions platinum, silver, and copper cause hypophosphorous acid and phosphorous acid to split off hydrogen, the final products being phosphorous acid and orthophosphoric acid respectively, while the intermediate products are presumably metaphosphorous acid and metaphosphoric acid respectively. The reactions may be written

$$H_3PO_2 + H_2O = HPO_2 + H_2O + H_2 = H_3PO_3 + H_2.$$
$$H_3PO_3 + H_2O = HPO_3 + H_2O + H_2 = H_3PO_4 + H_2.$$

It is the natural thing to do to consider the production of formaldehyde from methyl alcohol in presence of platinum as a plain case of limited oxidation of methyl alcohol; but there seems now to be no question but that the real reaction[34] is undoubtedly a catalytic splitting of methyl alcohol into formaldehyde and hydrogen, the nascent hydrogen then reacting with oxygen, which latter therefore really acts as a depolarizer. It is probable, though not proved, that the oxidation of alcohol to acetaldehyde by osmium tetroxide[35] may involve a catalytic dissociation as a first step.

An interesting case of the splitting-off of hydrogen is the conversion of alcohol into acetic acid by means of hot soda-lime.

[33] Zeit. anorg. Chem., **64**, 59 (1909); **76**, 1 (1912); Zeit. phys. Chem., **91**, 199 (1916).
[34] *Cf.* LeBlanc and Plaschke: Zeit. Elektrochemie, **17**, 471 (1911).
[35] Hofmann: Ber. deutsch. chem. Ges., **46**, 1657, 2854 (1913).

The reaction is usually written $CH_3CH_2OH + NaOH = CH_3CO_2Na + 2H_2$; but Mr. Carroll has found that the reaction takes place in several stages:

$$CH_3CH_2OH + NaOH = CH_3CHO + NaOH + H_2 = CH_3CO + NaOH + 2H_2 = CH_3CO_2Na + 2H_2.$$

The caustic soda is the catalytic agent and the lime is practically inert at the temperatures in question.

The catalytic action of caustic alkalies accounts for the fact that hydrogen is so often evolved when organic compounds are fused with alkalies.[36] Thus oleic acid reacts with fused caustic potash to form potassium palmitate, potassium acetate, and hydrogen[37] according to the equation

$$C_{17}H_{33}CO_2H + 2KOH = C_{15}H_{31}CO_2K + CH_3CO_2K + H_2.$$

In order to cut down the reducing action of the nascent hydrogen it may even be necessary to add an oxidizing agent, as in the production of alizarine from anthraquinone sulphonic acid.

It hardly seems probable that this evolution of hydrogen should depend on the organic substance forming a second liquid layer with fused alkali, and yet, if not, we pass direct from catalytic action in heterogeneous systems to catalysis in homogeneous systems. In presence of cuprous oxide and alkali, formaldehyde gives off hydrogen[38] according to the equation $HCHO + NaOH = HCO_2Na + H_2$. This is clearly a catalytic splitting-off of hydrogen in presence of cuprous oxide. If we leave out the cuprous oxide the reaction becomes $2HCHO + NaOH = HCO_2Na + CH_3OH$. This is usually described as simultaneous oxidation and reduction, but it is really a case of dissociation, the nascent hydrogen reducing some of the formaldehyde to methyl alcohol. If we have present a substance which is reduced more readily than formaldehyde, such as sodium peroxide, the reaction is of course slightly different[39] and may be written:

$$2HCHO + Na_2O_2 = 2HCO_2Na + H_2O + H_2.$$

One does not like to talk about adsorption in homogeneous systems, and I think it would be undesirable to try to formulate

[36] Lassar-Cohn: Arbeitsmethoden für organisch-chemische Laboratorien, 264 (1903).
[37] Varrentrapp: Liebig's Ann., **35**, 196 (1840).
[38] Loew: Ber. deutsch. chem. Ges., **20**, 144 (1887).
[39] Frankforter and West: Jour. Am. Chem. Soc., **27**, 714 (1905).

now a theory of catalysis in homogeneous systems. I do wish, however, to call attention to part of what Nernst says about electro-striction of free ions.[40]

"It is very remarkable that the values for the change of volume of dissociated substances [dissolving in water] are extraordinarily small, being considerably smaller than the molecular volume of the solid substance. In some cases (*e. g.,* sodium carbonate, magnesium sulphate, zinc sulphate), they are even negative. It seems, therefore, that with increasing separation of ions, the change of volume has in general a marked tendency to fall off; this may be seen clearly from the exact measurements of Kohlrausch and Hallwachs. The effects hitherto observed can be explained most easily by supposing the *solvent water to be strongly contracted by the presence of free ions.* It is of interest to know that such a contraction might be expected from the electric charges of the ions; every electric liquid must suffer a contraction if, as usually happens, the di-electric constant increases with pressure. It is therefore natural to suppose that the observed contraction of water by dissolved electrolytes is due to the electrostatic field of the ions; the theory of this electro-striction by free ions has been developed by Drude and Nernst.[41] This electro-striction, which, according to firm physical principles, must always be present, causes therefore a contraction of the solvent; the increase in internal pressure thus brought about will not be distributed equally over the whole solvent, but, on the contrary, will be very great in the immediate neighborhood of each ion, where a strong potential gradient exists, and will fall off rapidly at a short distance away. In many respects, however, the solvent will behave as if it were evenly subjected to a higher pressure. In this way we arrive at a very simple physical interpretation of the internal pressure of solutions."

Kohlrausch[42] has also emphasized the idea that there is a shell of water round an ion, which differs to some extent from the rest of the water. Kohlrausch makes the following hypotheses: "About every ion moves an atmosphere of the solvent, whose dimensions are determined by the individual characteristics of the ion. The atmospheres of multivalent or compound ions differ

[40] Nernst: Theoretical Chemistry, 420 (1916).
[41] Zeit. phys. Chem., **15**, 79 (1894).
[42] Proc. Roy. Soc., **71**, 338 (1903).

from those of monatomic ions. Data are at present lacking for a more complete representation. The electrolytic resistance of an ion is a frictional resistance that increases with the dimensions of the atmosphere. The direct action between the ion and the outer portion of the solvent diminishes as the atmosphere becomes of greater thickness." One of the conclusions drawn by Kohlrausch from these premises and from the data is as follows: "I now come to the remarkable relationship between the mobility of the ions and their temperature coefficients. This first led me to seek a general explanation for the electrolytic resistance in the idea of a water atmosphere, in order to escape being compelled to explain this otherwise irreconcilable fundamental characteristic of the ions as a *deus ex machina*. Assuming as the single fundamental characteristic of each univalent monatomic ion the formation of a water atmosphere, which varies according to the nature of the ion, the mobility of the complex on the one side, and its temperature coefficient on the other, will be functions of the atmospheric formations, and therefore both quantities must hold functional relations to each other. We know too little of the molecular forces at present to attempt to describe this connection more exactly. But for the case in which the water shell is so thick that the ion exerts no force beyond it, the resistance to motion becomes simply a matter of water friction, which explains the fact that the most sluggish ions have nearly the same temperature coefficients as the viscosity. In the case of smaller aggregations, we must remain content with the fact that we have at least the possibility of a fundamental explanation."

If one admits the existence of special water films around the ions, all gradations between heterogeneous and homogeneous systems become possible. It opens up all sorts of fascinating possibilities; but now is not the time to discuss them.

Coming back to definite cases of contact catalysis, there are several instances which perhaps call for a word of comment. In all such cases as the decomposition of arsenic hydride, etc., the reaction takes place practically completely at the surface of the containing vessel and not in the mass of the gas. To take a single case, the rate of decomposition of antimony hydride is accelerated by the presence of metallic antimony.[43] If we assume

[43] Stock and Guttmann: Ber. deutsch. chem. Ges., **37**, 901; Bodenstein: 1361 (1904).

that antimony adsorbs or tends to make antimony vapor condense upon it, the catalytic action follows at once. Another case which also belongs in this class is the decomposition of silver oxide into silver and oxygen. Lewis[44] has shown that this reaction is accelerated by silver. At first sight this looks like the catalytic decomposition of one solid by another; but it seems impossible that the silver should act at a distance. It is probable, therefore, that we are really dealing with the decomposition of silver oxide vapor, in which case the reaction differs only in degree from the decomposition of stibnine.

Taylor and Hulett[45] have shown that the decomposition of mercuric oxide is facilitated by finely-divided platinum, ferric oxide, manganese dioxide, and cadmium oxide, but not by stannic oxide or alumina. While this is undoubtedly an adsorption phenomenon, the data are not sufficient to enable one to account satisfactorily for the details.

The general conclusions to be drawn from this paper are:

1. Only those substances which are adsorbed by a solid are catalyzed by it.

2. While the catalytic action of solids may be solely the result of the increased surface concentration in some cases, this is not always the only factor.

3. A solid catalytic agent may be considered as equivalent to a solvent and may therefore displace the equilibrium.

4. As a result of selective adsorption we may get different reaction products with different catalytic agents.

5. A catalytic agent tends to produce the system which it adsorbs the more strongly.

Cornell University.

DISCUSSION.

PRESIDENT C. G. FINK[1]: I am very much interested in the papers presented by Prof. Bancroft. I note that Prof. Bancroft omitted any allusion to a paper of mine published ten and a half years ago. (See Zeit. Phys. Chem., vol. **60**, pp. 1-69 (1907).)

[44] Zeit. phys. Chem., **52**, 310 (1905).
[45] Jour. Phys. Chem., **17**, 568 (1913).

[1] Director of Research, Chile Exploration Co., New York City.

As Prof. Bancroft says, "Your paper on adsorption of sulphuric acid anhydride is very interesting, but at the time it appeared it did not fit in with the Van't Hoff ideas. You published it too soon. Why didn't you wait?"

On page 462, Prof. Bancroft says: "Since contract catalysis involves adsorption, a decrease in the adsorption will mean a decrease in the reaction velocity, other things remaining the same." "Decrease in adsorption" should be modified to read "a decrease in the adsorption of the reacting components," in contradistinction to the reaction product.

A decrease in the adsorption of the product SO_3 *increased* the velocity. That is, if you can remove the SO_3 in some way or other from the surface of the platinum as fast as it is formed, the reaction velocity will increase. The velocity of the formation of SO_3 from SO_2 and O_2 is inversely proportional to the square root of the concentration of the SO_3. The greater the SO_3 concentration and the thicker the SO_3 film through which fresh quantities of SO_2 and O_2 must pass, the slower the reaction.

On page 463, under caption 10, Dr. Bancroft says: "Sulphur trioxide decreases the rate at which sulphur dioxide and oxygen react." This is so; but I think No. 10 could be fitted in with No. 1 without any harm .

G. C. GIVEN[2]: With reference to the catalytic action of a copper cathode and an amalgamated mercury cathode, respectively, on the reduction of nitric acid, I cannot quite reconcile the adsorption theory to that; nor can I think it is due to a difference in over-voltage. With a copper cathode nitric acid is reduced to ammonia, with an amalgamated mercury cathode it is reduced to hydroxylamine. I would like to have that cleared up.

F. E. CARTER[3]: I am afraid I am not a specialist in the subject of catalysis, but I would like to ask Dr. Bancroft if I am correct in seeing a contradiction in the two statements—"A catalytic agent tends to produce the system which it adsorbs the more strongly" (conclusion 5, page 490) and "Since the presence of the reaction products will decrease the rate at which the reacting substances will diffuse to the catalytic agent and since the

[2] Chemist, Atlas Powder Co., Tamaqua, Pa.
[3] Physical Metallurgist, Baker & Co., Newark, N. J.

reaction products may decrease the adsorption of the reacting substances, the reaction products may decrease the reaction velocity if they are not removed" (conclusion 8, page 463). Personally, I feel that if the products are not adsorbed they will readily escape from the film of high concentration in which the reaction is taking place, and more of the products will continually be formed.

It seems to me that the terms "factors" and "products" would be simpler and more convenient in use than "reacting substances" and "reaction products." There is one other point. We know from thermo-dynamics that in gaseous reactions where the volume of the "products" is less than the volume of the "factors," increase in pressure shifts the equilibrium so that more of the "products" are formed; the Haber process of synthetic ammonia is an example of this. Now, do you get the same thing in catalysis? My idea of catalysis is that when adsorption takes place, you get a highly concentrated but still gaseous film around the solid material; from analogy with the effect of pressure change on gaseous reactions, I would expect that the catalyst would not shift the equilibrium where the volume of the "products" is the same as the volume of the "factors," and indeed that the equilibrium might be shifted in either direction according to the relative volumes of "factors" and "products." Is this the experience in investigations on catalysis?

President C. G. Fink: That is a very good point. If the SO_2 were more strongly adsorbed than the SO_3, we would observe a different (apparent) reaction velocity than in our case, in which the SO_3 was more strongly adsorbed than the sulphur dioxide.

Prof. W. D. Bancroft: In a more extended series of papers, I have given a number of literature references which are omitted here, and among those I have given references to some work done in Bodenstein's Laboratory. I did not give any reference, I am sorry to say, to Mr. Fink's paper. He gave me a copy of it the other day, and it should have been mentioned, because he had much of the theory worked out perfectly clearly more than ten years ago. The only explanation I have for forgetting the paper is that undoubtedly I did not know at that time how good it was.

As a matter of fact, if Mr. Fink had stayed in theoretical work, instead of taking up technical work, I think our knowledge of colloid chemistry would have been further along. People were not ready for what he told us then.

Now, as regards the ammonia and the hydroxylamine, it is unquestionably not a question of voltage alone. I do not know the answer now, but it seems to me quite possible that hydroxylamine is adsorbed by the mercury, and ammonia by the copper. At any rate, that is the line along which I would attack that problem. As far as I know, no other explanation has been offered at the present time. I wrote a long article years ago, calling attention to the facts and pointing out that the result is the same whether the reaction takes place "chemically or electrochemically."

The other statement in regard to the adsorption of the products does not seem to me to show any contradiction. If a given reaction product is adsorbed very strongly, the reaction will tend to run in that direction, just as when you keep vapor pressure or concentration very low, the reaction will run to an end. But that does not prevent the fact that the reaction may stop in case you do not remove the reaction product. We know that sulphur trioxide, if not removed, cuts down the reaction velocity. Let us take a case where the adsorption is so marked that we see clearly what happens. Platinum causes the decomposition of amygdalin, and the prussic acid formed poisons the platinum and brings the reaction to a standstill so soon as enough prussic acid is formed. Similar phenomena occur in enzyme reactions. An enzyme will not necessarily convert an indefinite amount of a given substance. In some cases there must be an approximate relation between the amount of enzyme and the amount of reaction product, or the reaction does not run to an end. These are cases where there is marked adsorption and where the reaction product has cut down the rate at which the reaction takes place, just as sulphur trioxide does in the contact sulphuric acid process.

I am not quite sure that I understand the bearing of the question on the effect of pressure. We know that in simple cases, where there is no reaction, the amount of adsorption of a gas increases with increasing reaction velocity if you increase the

pressure under which your gases are fed in. We know that in the case of the Haber process, Haber thought that an increase of pressure was desirable because the reaction takes place with contraction of volume. That is right, if one is dealing with reversible equilibrium. I am inclined to think, however, that the effect of the higher pressure in the Haber process is due but slightly to that cause, and is due largely to the fact that higher pressure increases the concentration in the adsorption film. That is immaterial to the theory, but I am glad it was brought up, since I had not thought of that side of that possibility.

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